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UNIVERSITY OF ALBERTA

INSTRUMENTAL AND SENSORY ANALYSES
OF PROCESSED SASKATOON BERRY JUICE

by

SUSAN ELIZABETH LUTZ ©

A THESIS
SUBMITTED TO THE FACULTY OF GRADUATE STUDIES AND RESEARCH
IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE DEGREE OF
DOCTOR OF PHILOSOPHY
IN
FOOD SCIENCE

DEPARTMENT OF FOOD SCIENCE AND NUTRITION

EDMONTON, ALBERTA
FALL, 1994

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DEDICATION

This thesis is dedicated to :

my mother, Beverly Shimmin, and my grandmother, 'Bobby', (UBC alumni)

for their encouragement to pursue my education to the highest level,

and

Dr. Gary Reineccius for the inspiration to pursue my studies

in the field of flavour chemistry.

ABSTRACT

Saskatoon berries are juiced for manufacture of wine, jellies, and syrups. This study investigated the effects of four processing methods: cold (C); pectinase enzyme (P); hemicellulase + pectinase enzyme (HP); and hot (H) on saskatoon juice quality using instrumental and sensory analyses.

For HP, H, P, and C juices, yields were 56%, 46%, 44%, and 40% respectively. C, P, and HP juices were yellower and lighter than H juice. Glucose was higher ($p \leq 0.05$) in H juice than in C, P, and HP juices; fructose was similar in all juices. Malic, citric, quinic, fumaric, and ascorbic acids were found in saskatoon juices. GC/MS analysis determined 48 volatile constituents. There were significant differences in volatiles among juices especially for benzaldehyde (high in H juice), benzyl alcohol (HP > P > H > C), and benzoic acid (high in P and HP juices). Further experiments showed: 1) low benzaldehyde, benzyl alcohol, and benzoic acid levels in seedless juice; 2) heat and water activated two enzyme systems in juices with seeds producing benzaldehyde and benzyl alcohol; and 3) benzaldehyde, derived from amygdalin in saskatoon berry seeds, is oxidized to benzoic acid.

A trained QDA panel (n=6) evaluated eleven characteristics in juices. C juice was prune brown coloured, cloudy, musty, pruney, sour, and dry. P and HP juices were like C juice with additional slightly almondy and strong medicinal notes. H juice was black cherry coloured, clear, fresh/fruity, almondy, sweet (in odour and taste), and dry. Two consumer free-choice profiling (FCP) panels (2 X n=24) evaluated juices. QDA and two FCP panel data were analysed by generalised Procrustes analysis (GPA). Panel consensus configurations were similar. For each juice, FCP and QDA panel descriptions were similar. FCP panels liked H juice more ($p \leq 0.05$) than HP, C, and P juices.

GPA determined relationships among instrumental and QDA sensory data including: prune brown colour with 'L' and 'b' values; dark cherry red colour with 'L' and 'a' values; sugars with sweetness; almondy with benzaldehyde; sweet odour with benzaldehyde and phenyl ethanol; fresh fruity with butyl butanoate; medicinal with benzyl alcohol; musty with linalool oxide; and strong flavour with benzaldehyde, phenyl ethanol, butyl butanoate, and benzyl alcohol.

Thus, processing method significantly affected the chemical composition and sensory profile of saskatoon berry juice.

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LIST OF ABBREVIATIONS

ANOVA	analysis of variance
C	cold processed (juice)
FCP	free-choice profiling
GC	gas chromatography
GPA	generalised Procrustes analysis
H	hot processed (juice)
HP	hemicellulase + pectinase processed (juice)
HPLC	high performance liquid chromatography
MS	mass spectroscopy
NS	no seeds (juice)
P	pectinase processed (juice)
PC1/2	principal coordinate one / principal coordinate two
QDA	quantitative descriptive analysis
SEM	standard error of the means
SNK	Student-Neuman-Keuls (difference test)
SW	seeds + water (juice)

Chapter 1 General Introduction

A commercial variety of saskatoon shrubs, *Amelanchier alnifolia* cv. Smoky, is being cultivated in Alberta. Current saskatoon acreages may yield over 500,000 kg of fruit by the late 1990's (Hausher, 1988). This fruit will provide opportunity for development of a value-added processed saskatoon berry industry. Saskatoon berries are processed into juice to manufacture popular products such as wine, jellies, and syrups. High quality juice is essential for the manufacture of quality finished products. Cold, hot, and enzyme assisted juicing processes are currently used industrially for fruits such as apples and blueberries. Researchers (Wolfe and Wood, 1971; Mazza et al., 1978; Mazza, 1979) have studied the effect of heat and enzyme applications on saskatoon berries to produce laboratory pressed juices. Quality parameters investigated in these studies were limited to yield, acids, and sugars. Mazza (1980) found that 45.4% of the volatile aroma constituents, as determined by gas chromatography (GC) headspace analysis, were lost when saskatoon berries were juiced. Subsequently, using a distillation technique, Mazza and Hodgins (1985) determined that benzaldehyde was a major aroma component. Majak et al. (1981) found that cyanogenic glycosides, present in saskatoon berries, may lead, through crushing and heating, to benzaldehyde. No other aroma components have been identified in saskatoon berries and published sensory evaluations of saskatoon juice quality are lacking.

Increased availability of saskatoon berries for processing into value-added products has created the need for information on the effect of commercial processing techniques on the quality of saskatoon juice. The identification of volatile aroma constituents and a sensory profile, currently lacking, are required to assess saskatoon juice quality.

1.1. Saskatoon Berry Industry

Native saskatoon shrubs, *Amelanchier alnifolia*, grow from the southern Yukon and Northwest Territories of Canada to the northern United States (Harris, 1972). The saskatoon berry is a rosaceous fruit or pome and is closely related to apples, pears, and quinces. The berries, a major source of fruit for the indigenous people and settlers that followed, continue to be popular with western Canadians. They are eaten fresh or made into pemmican, pies, jams, jellies, syrups, and wine.

Development of commercial saskatoon varieties was initiated by Agriculture Canada at the Beaverlodge Research Station in 1918 (Davidson, 1986). In 1953, the Smoky cultivar was selected as the most suitable for commercial production. Currently, both Agriculture Canada and Alberta Agriculture, Food and Rural Development research centres are evaluating other saskatoon varieties such as Pembina, Northline, Honeywood, Forestburg, Porter, and Thiessen for commercialisation.

Published research related to commercialisation of saskatoon production has included studies on: propagation (Bishop and Nelson, 1980; Nelson and Bishop, 1980), fruit ripening (Olson and Steeves, 1982; Green and Mazza, 1986), and cyanide potential (Majak et al., 1981). Majak et al. (1981) determined that cyanogenic glycosides in saskatoon foliage and fruit could be hazardous to browsing ruminants.

The first commercial acres of saskatoons were planted in Alberta in 1972; by 1987 there were 363 acres which were predominantly cv. Smoky (Hausher, 1988). Four year old saskatoon bushes may yield a fruit harvest of 225 kg/acre, while mature (seven year) bushes may produce more than 1000 kg/acre. In 1987, only 15% of planted saskatoon bushes were mature. As more bushes are planted and mature, berry yield could exceed 500,000 kg by the end of this century.

In 1987, 54% of saskatoon berries harvested were sold fresh mainly through U-pick operations (Hausher, 1988). The remainder were frozen (19%), processed into wine (15%), or condiments (12%) such as jams, jellies, and syrups. Success of the U-pick fresh saskatoon market has depended on proximity to urban centres. New saskatoon plantings may not be located near cities; thus increased yields in the 1990's will saturate the U-pick market. Increased saskatoon production will provide opportunity for development of the value-added processed saskatoon berry industry.

Fresh fruit may be frozen for manufacture into value-added products. The Peace Country Fruit Producers Co-operative operates an "individually quick frozen" plant for saskatoon berries in Hythe, Alberta. Other berries are blast frozen at commercial freezer facilities. Whole fresh or frozen saskatoon berries may be processed into products such as pemmican, jams, and pies. Berries must be juiced for the manufacture of wine, jellies, and syrups. The latter products are currently more prevalent in the domestic marketplace and are expected to expand into export markets.

1.2. Fruit Juice Processing

Commercially, apples have been processed into juice since the early 1900's. Initial research related to juice production focussed on: 1) fruit soundness and maturity, 2) physical pressing, filtering, and deaerating, 3) preventing oxidative changes, and 4) heat preservation techniques (Moyer and Aitken, 1980). In the last two decades, research into and subsequent industrial use of enzymes and heat treatments has increased yields and improved efficiencies in apple juice production (Boyce, 1986). Research on saskatoon juice processing is lacking; thus studies of the effect of enzyme and heat treatments on saskatoon juice production are warranted.

1.2.1. Pressing and Filtering

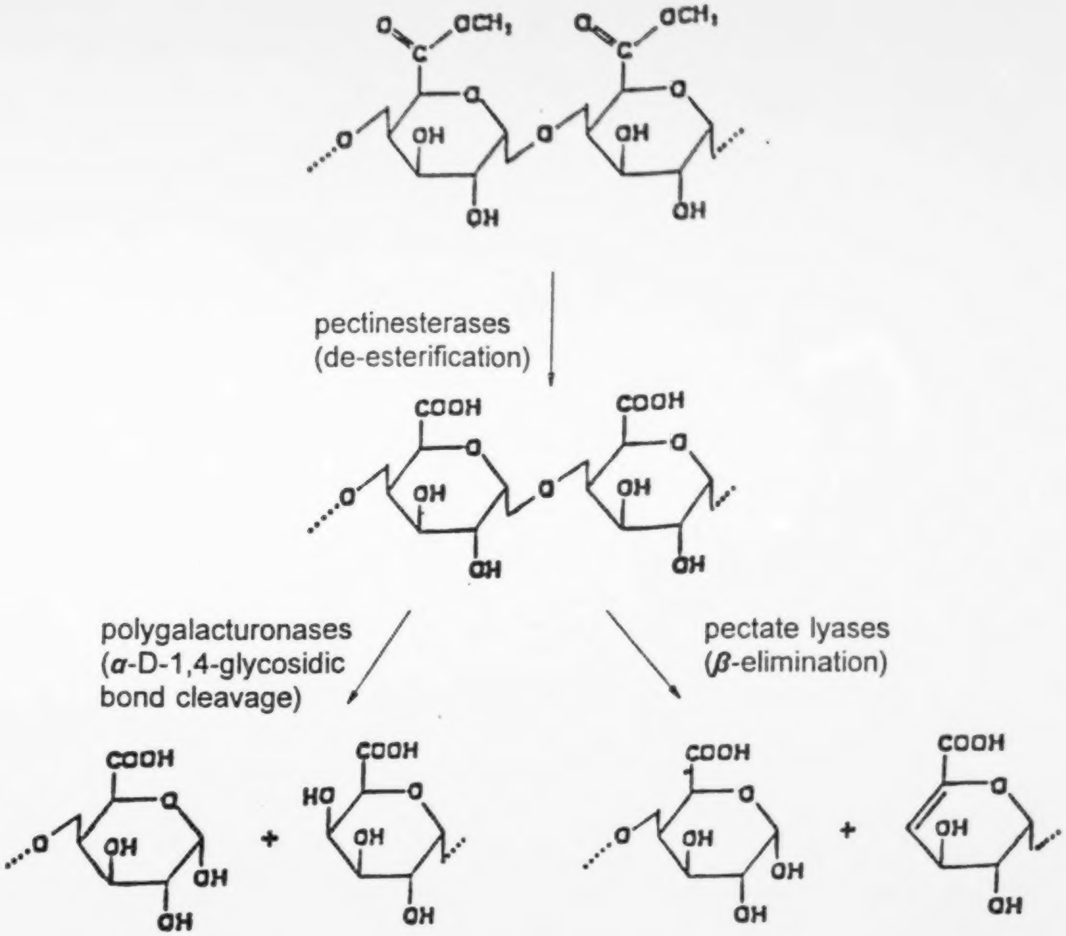
Fruit juices are processed commercially via pressing and filtering. A variety of industrial presses including hydraulic, pneumatic, continuous screw, and continuous plates are available. Filters are usually stainless steel support plates which have been precoated with a filter medium, commonly diatomaceous earth. Continuous centrifuges may also be used without or with filters to clarify fruit juice.

Published literature on saskatoon berry juicing is limited to laboratory scale pressing and filtering (Mazza et al., 1978; Mazza, 1979). Mazza et al. (1978) juiced one kg batches of saskatoon berries using a Carver laboratory press and Whatman filter paper.

1.2.2. Enzyme Processing

Processed saskatoon juice contains suspended pectinaceous material. The soluble pectin increases viscosity, slows filtration, and forms stable particles which cloud the juice. It is difficult to efficiently filter pressed juice unless it has been pretreated with enzymes to reduce viscosity and particle size.

Pectins are high molecular weight (30,000 - 100,000) polysaccharides composed mainly of galacturonic acid units in α-1,4 linkages. Three types of enzymes may degrade pectin structures (Figure 1.1). Pectinesterases deesterify methyl esters of polygalacturonates. α-D-1,4-glycosidic bonds may

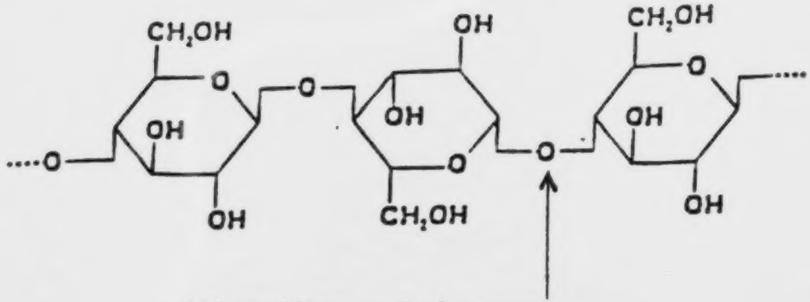

OCH3
OCH3
O
C
OH
pectinesterases
(de-esterification)
COOH
polygalacturonases
(α-D-1,4-glycosidic
bond cleavage)
pectate lyases
(β-elimination)
HO
+

[Adapted from Wong (1989)]

Figure 1.1. Pectic enzyme degradation of pectin.

be cleaved with polygalacturonases which catalyze hydrolysis of non esterified units, or via pectate lyases which catalyze degradation through *β*-elimination. Enzyme functionality is temperature dependent. Time/temperature relationships for enzyme degradation in commercial apple juice processing vary from 6-8 hr at 16°C to 1-2 hr at 54°C (Boyce, 1986).

Enzyme mixtures may also be used prior to pressing to degrade pectin and cellulose. Cellulose is the main structural material of plant cell walls. It is composed of repeating cellobiose units, each consisting of two β-1,4 linked glucose units (Figure 1.2). β-glucosidases and β-1,4 gluconases cleave internal β-1,4 linkages or remove glucose units from the end of the chain.



β-glucosidase or β-gluconase
splitting of β-1,4 linkages

Figure 1.2. Enzyme degradation of cellulose.

When enzymes are used in commercial processing, the fruit is ground, heated and pectic enzymes and cellulases are added for 30-60 min at 15°-30°C (Boyce, 1986). An enzyme treated filtered mash can yield an extra 6-10% juice (Boyce, 1986). A study of blueberry juices gave yields of 58.4% for cold pressing and 70.9% for enzyme assisted pressing (Yang, 1984). The viscosity of cold pressed juice decreased from 17cp to 12cp for enzyme processed juice (Yang, 1984).

Mazza et al. (1978) reported preliminary trials of saskatoon juice extraction using a hydraulic press and a continuous screw pulper-finisher. Both methods gave low yields and juices that tended to gel. Further work, using laboratory equipment, included addition of varying levels of pectolytic enzymes to the mash (Mazza et al., 1978; Mazza, 1979 ; Mazza, 1980) and heating the mash to 63°C prior to pressing and filtering. Differences in juice yields due to enzyme use were not observed. The laboratory hydraulic press used in these experiments did not simulate the juicing action of commercial screw pulper finishers. Thus, saskatoon juicing studies using a pilot plant screw pulper finisher together with enzyme and heat treatments should be conducted to more accurately determine the effect of enzymes on the yield of commercially produced saskatoon juice.

1.2.3. Heat Processing

Apple mashes may be heated to optimize enzyme activity and/or coagulate colloidal material. Temperatures in excess of 80°C may be applied to pressed juices to coagulate colloidal material (Moyer and Aitken, 1980). The heated juice is cooled rapidly and centrifuged to remove the coagulated particles.

Whole fruit may be heated prior to pressing to increase yield, total acidity, and soluble solids, and to stabilize colour. Heated blueberry (Yang, 1984) and cherry (Tressler et al., 1980) juices had increased yields (2-6%) and clarity, and deeper purple/red colours than unheated juices. Mazza et al. (1978) observed increases from 5.7% to 9.5% in soluble solids content of juices when enzyme treated saskatoon berry mashes were heated from 49°C to 93°C. Yang (1984) and Mazza et al. (1978) also noted slight increases in total acidity of heated and enzyme processed blueberry and saskatoon juices, respectively.

1.3. Flavour Chemistry of Fruits

The flavour of fruits is due to non-volatile constituents such as sugars, acids, and phenolic compounds as well as volatile organic compounds. Although research on saskatoon berry flavour is lacking, studies on other fruits indicate that flavour variations within a fruit may be attributed to varietal differences, horticultural practices, maturity and storage conditions. Paillard (1967) showed that different apple varieties contain similar volatile constituents but their proportions vary. McIntosh apples grown with nitrogen fertilizers contained greater quantities of volatiles which intensified flavour more than those grown without added nitrogen fertilizers (Somogyi et al., 1964). Environmental conditions such as plentiful water and low temperatures may decrease flavour (Heath and Reineccius, 1986). During tree ripening, initially volatile alcohols and later esters, especially low molecular weight ethyl esters, increase as fruit matures (Dirinck et al., 1984). Harvested immature peaches developed only 20% of the benzaldehyde normally found in tree ripened fruit (Do et al., 1969). Frozen storage may influence fruit quality; however, frozen saskatoon berries stored (-20°C) up to 6 mo showed no substantial changes in moisture, sugar content, volatiles, or colour (ASCHRC, 1988).

1.3.1. Non-volatile Flavour Constituents

The types and quantities of sugars and acids present significantly affect the flavour of pome fruits. Sugars make up 5 - 16% while acids account for 0.4 - 1.0% of the fresh fruit weight for pomes (Hulme and Rhodes, 1971). Sugars common to pomes are fructose, glucose, and sucrose which provide sweetness depending on their presence and proportions. Acids present in pome family fruits include malic, citric, and quinic acids. Malic and citric acids contribute to tartness while quinic acid provides astringency. Ascorbic acid, also present in fruits and berries, does not significantly contribute to flavour. During ripening the sugar:acid ratio increases and alters the flavour of maturing pome fruits.

1.3.2. Biogenesis of Fruit Aroma

Most aroma constituents of fruits develop during ripening when plant respiration increases and the fruit metabolism changes to catabolism. Lipids, free amino acids, and carbohydrates are enzymatically converted to flavour volatiles which, in turn give a variety of volatile constituents that contribute to flavour, or enter inter-related metabolic pathways and are further converted to other aroma compounds (Salunke and Do, 1976).

Lipids comprise < 0.6% of many fruits (USDA, 1975). Lipids and fatty acid flavour precursors have been identified in apples. Galliard (1968) found 880 mg of lipids in 1000 g of apple pulp. Linoleic acid accounted for 53% and hexadecanoic acid for 31% of the total fatty acids identified. Fatty acids from lipids are metabolised by various pathways to give aldehydes and ketones as primary products. Further reactions generate acids, alcohols, lactones and esters (Figure 1.3). Examples of these reactions, important to fruit flavours, include: β-oxidation of linoleic acid to produce esters; hydroxyacid cleavage to give lactones in peaches; and lipoxygenase enzyme oxidation of linoleic and linolenic acids to produce aliphatic esters, alcohols, acids, and carbonyls (Jennings, 1967; Tressl and Jennings, 1972; Maga, 1976;).

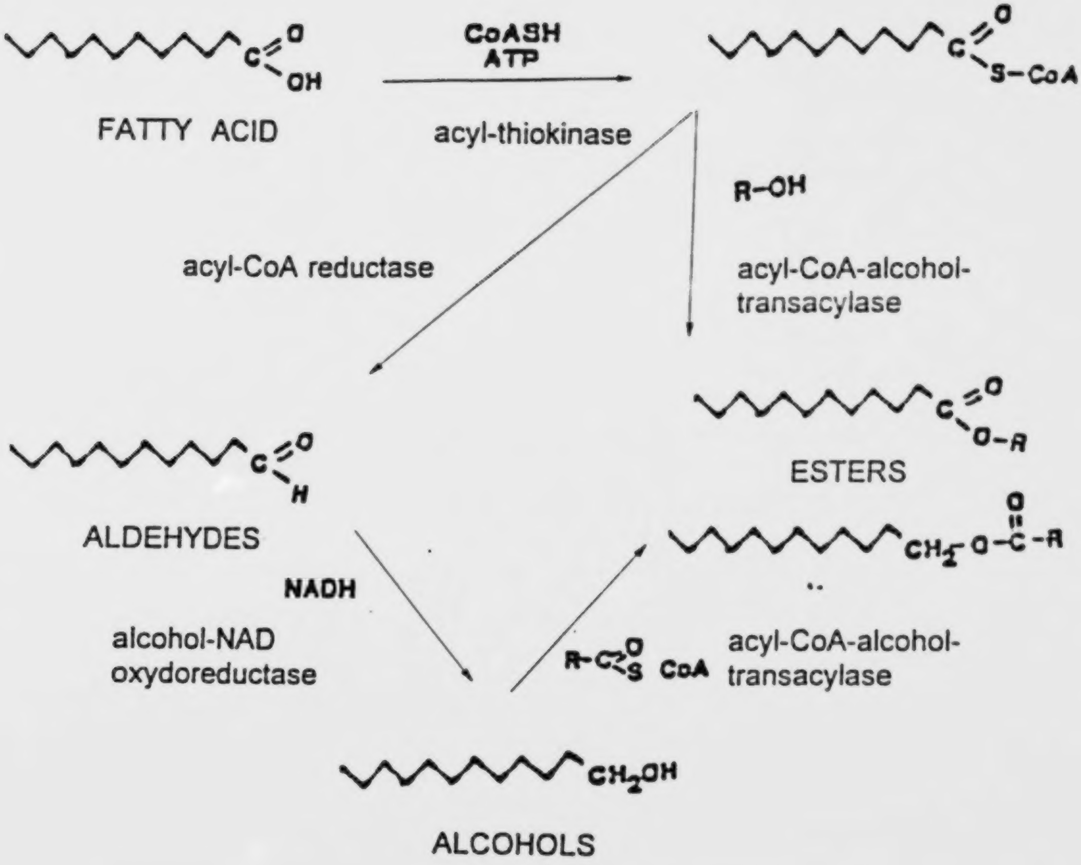
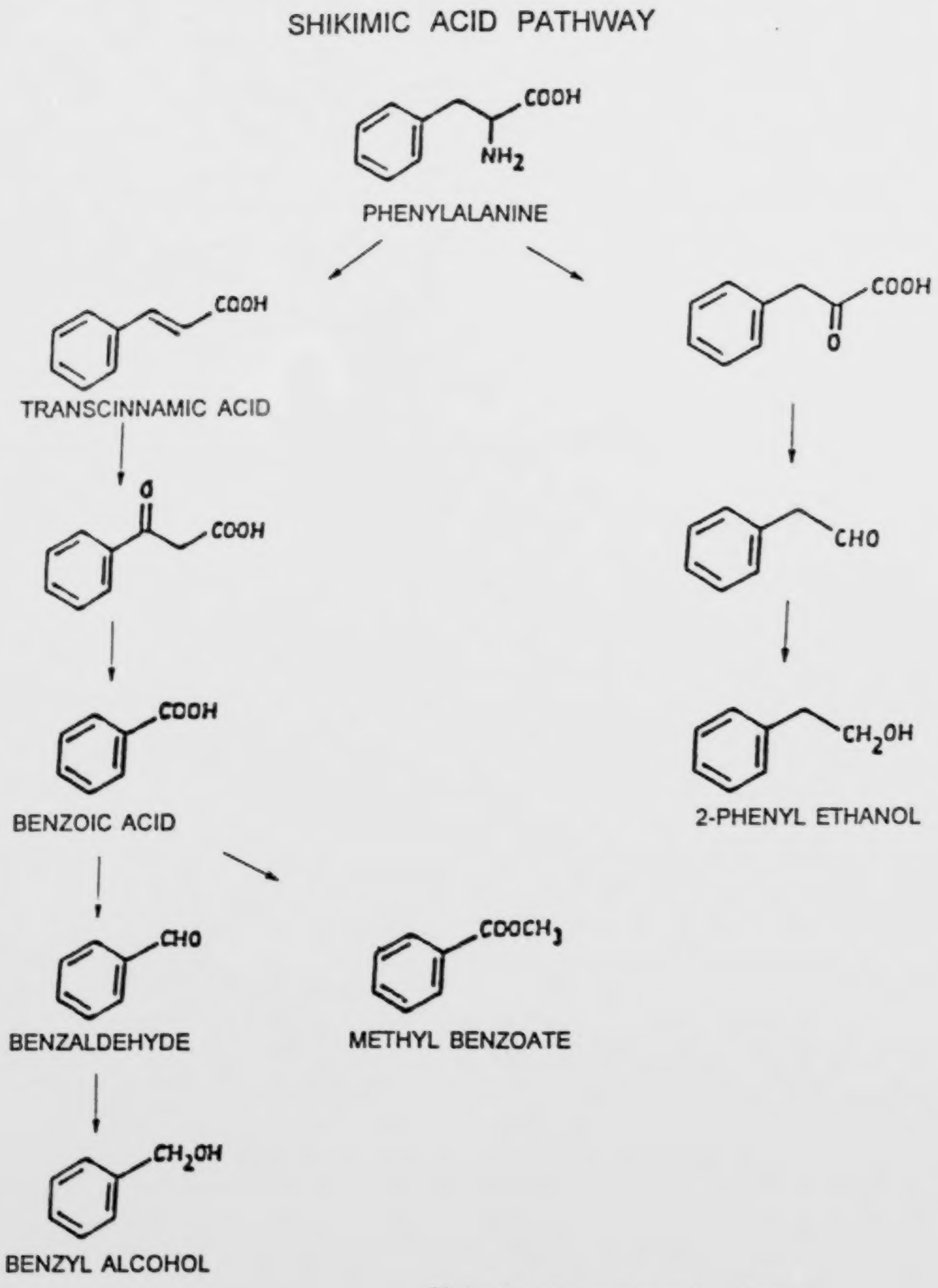

FATTY ACID
CoASH
ATP
acyl-thiokinase
R-OH
acyl-CoA reductase
acyl-CoA-alcohol-transacylase
ESTERS
ALDEHYDES
NADH
alcohol-NAD oxydoreductase
acyl-CoA-alcohol-transacylase
CoA
ALCOHOLS

Figure 1.3. Reaction pathway of fatty acids to esters.

Esters, aliphatic and branched alcohols, acids, and carbonyls may also arise from amino acid metabolism. Aromatic amino acids such as tryosine and phenylalanine are metabolised via the Shikimic acid pathway to produce aromatic acids, aldehydes, esters, and alcohols as shown in Figure 1.4 (Pyysalo, 1976).


SHIKIMIC ACID PATHWAY
COOH
NH2
PHENYLALANINE
COOH
TRANSCINNAMIC ACID
COOH
O
O
COOH
COOH
BENZOIC ACID
CHO
BENZALDEHYDE
COOCH3
METHYL BENZOATE
CH2OH
BENZYL ALCOHOL
CHO
CH2OH
2-PHENYL ETHANOL

[Adapted from Pyysalo (1976)]

Figure 1.4. Suggested derivation of aromatic compounds. (Named compounds have been identified in berries.)

Carbohydrates, the major constituent of fruits, may be metabolised to organic acids, esters, alcohols, aldehydes, and terpenes. Oxygenated terpenes, which provide the characteristic flavour of citrus fruits, are also present in small quantities in apples and berry fruits as α-terpineol and linalool (Paillard, 1990).

1.3.3. Aroma Constituents of Related Fruits

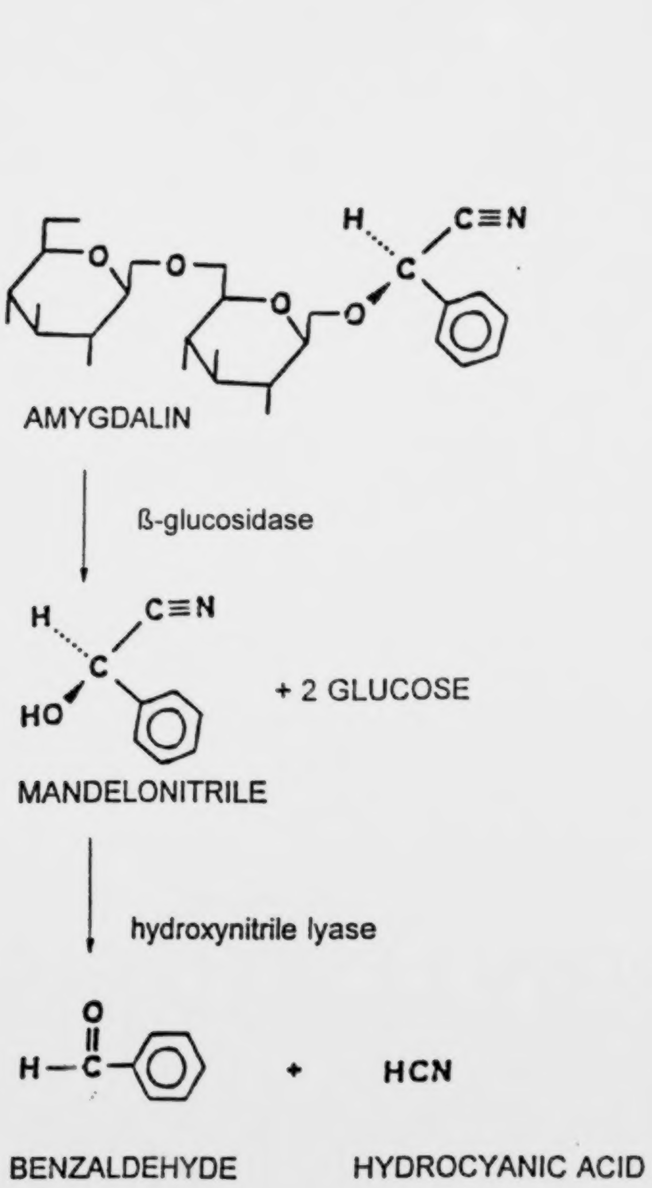
The rosaceae family, to which saskatoon berries belong, comprises apples, pears, quinces, strawberries, raspberries and blackberries. Other northern berries with similar aroma constituents include cranberries, blueberries, bilberries, currants, arctic bramble, cowberries, sea buckthorn, and cloudberries. These pomes or berries typically do not have a single character impact aroma constituent, rather their odour depends on a combination of aroma constituents which vary in quantity in specific fruits.

Esters are the most prevalent category of volatiles in pome and berry fruits. Esters with two to eighteen carbons have been reported in apples (Paillard, 1990). High molecular weight esters, such as ethyl hexadecanoate, are more common in fruits with mild odours, such as pear and quince, but have also been found in apple, blackberry, and blueberry aromas (Paillard, 1990; Honkanen and Hirvi, 1990).

Alcohols form the second largest group of volatile constituents in pome and berry fruits. Alcohols identified in pomes and berries include hexan-1-ol, butan-1-ol, (E) hex-2-en-1-ol, and (Z) hex-3-en-1-ol (Paillard, 1990; Honkanen and Hirvi, 1990). (E) hex-2-en-1-ol is one of three character impact compounds in bilberries (von Sydow and Anjou, 1969).

Terpenes are usually present in much lower quantities in berries than in citrus fruits where they significantly impact aroma. Linalool and α-terpineol are found in most pomes and berry fruits (Paillard, 1990). α-Terpineol, found in unusually high levels in cranberries, has been identified, along with several compounds, as characteristic of cranberry aroma (van Straten, 1981).

Aromatic compounds such as benzyl alcohol, phenylethanols, benzaldehyde, and benzoic acid are found in berries and stone fruits such as cherries, peaches, apricots, and plums. Broderick (1975) reported that benzaldehyde, a character impact aroma constituent of cherries, was derived from enzymatic hydrolysis of amygdalin glucoside present in the stone. Hydrolysis of amygdalin to mandelonitrile and its subsequent conversion to benzaldehyde and hydrogen cyanide is shown in Figure 1.5. Because it is very unstable conversion of mandelonitrile to benzaldehyde can occur spontaneously. Working with apricot pulp, Salles et al. (1988) determined that benzyl alcohol and 2-phenylethanol were glycosidically bound aromatic alcohols and linalool was a glycosidically bound terpene.


AMYGDALIN
ß-glucosidase
MANDELONITRILE
+ 2 GLUCOSE
hydroxynitrile lyase
BENZALDEHYDE
+
HCN
HYDROCYANIC ACID

[Adapted from Wong (1989)]

Figure 1.5. Derivation of benzaldehyde from amygdalin.

1.3.4. Effect of Processing on Aroma Constituents

Crushing/grinding fruit to create juice provides potential for natural enzymes present in the fruit to come in contact with their substrates and makes water available for the many enzyme mediated reactions. Schreier et al. (1978) found that esters were hydrolysed and C_6-aldehydes produced when apples were crushed. Glycosidically bound linalool furan oxides and α-terpineol were released during pureeing of apricots (Chairote et al., 1981).

Pectolytic and cellulolytic enzyme addition may influence volatile constituents. With apple juices, Schreier et al. (1978) determined that enzyme treatments affected the formation of C_6-aroma compounds; pectolytic enzymes preserved C_6-aldehydes, and cellulolytic enzymes encouraged reduction of the C_6-alkanals to corresponding alcohols.

Heat enhanced release of glycosidically bound terpenes and aromatic alcohols in apricot pulp (Chairote et al., 1981) and cherries (Schmid and Grosche, 1986). Heat also oxidized monoterpenes to form oxides such as linalool oxides as shown in Figure 1.6 (Crouzet et al., 1983).

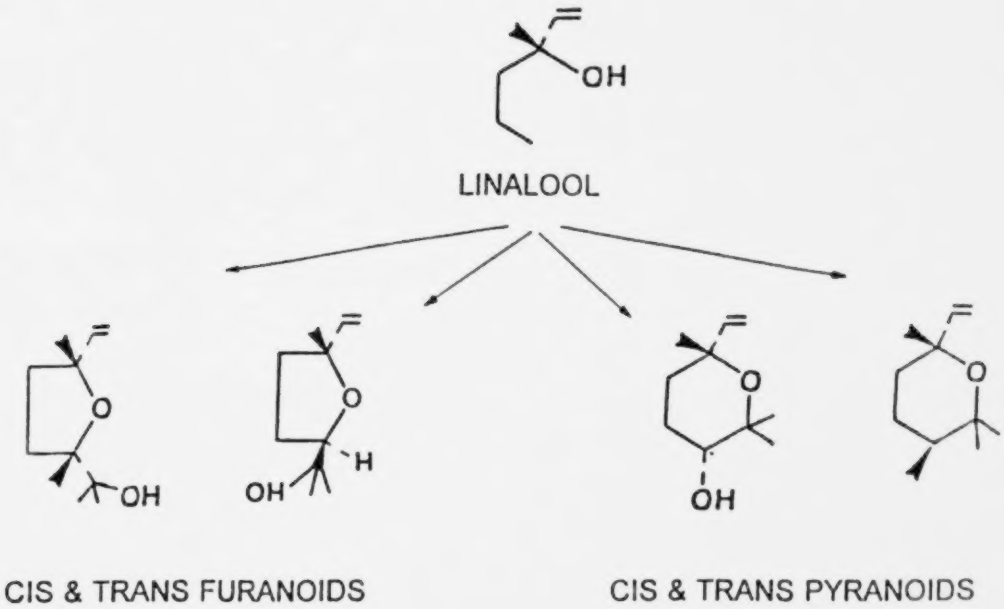


Figure 1.6. Formation of linalool oxides from linalool. (Crouzet et al., 1983)

1.3.5. Flavour Constituents of Saskatoon Berries

Sugar and acid profiles of saskatoon berry cultivars, differing in maturity, from various regions of Alberta have been reported by Wolfe and Wood (1971), Panther and Wolfe (1972) and Green and Mazza (1986). The major sugars in saskatoon berries were fructose and glucose; the amount of sucrose was too low for accurate quantitation (Wolfe and Wood, 1971). Wolfe and Wood (1971) determined that malic, citric, and oxalic acids in saskatoon berry juices were influenced by fruit maturity. Malic, oxalic, and citric acids were noted prior to saskatoon berry maturity; at maturity, oxalic acid was absent, citric acid had decreased, but malic acid was present. Ascorbic acid was absent possibly due to an ascorbic acid oxidizing system in saskatoon juice (Panther and Wolfe, 1972).

The sugar:acid ratios of saskatoon berries increased with maturity (Green and Mazza, 1986). As berries, cv Smoky, changed colour, from red to purple with red tinges and then to dark purple, sugar:acid ratios increased from 29 to 41 and to 65, respectively. Sugar caused the increase in the ratio; the very small acid content did not change significantly over time.

Volatile aroma constituents in fresh and processed saskatoon berries were quantified by GC headspace analysis (Mazza, 1980). The area under the GC peaks of juiced berries was 45.4% lower than that for whole berries (Mazza, 1980). Processing the juice into jelly decreased the area by an additional 86.5%. This study (Mazza, 1980) supports earlier findings (Mazza, 1979) which concluded that saskatoon jelly had a weak flavour.

Using a distillation technique with capillary GC and mass spectroscopy (MS), Mazza and Hodgins (1985), identified benzaldehyde as a major aroma component of saskatoon berries. The authors also used a spectrophotometric AOAC method requiring crushing, heating, and adding methanol to determine 168 mg benzaldehyde per kg saskatoon berries. In contrast, blueberries and raspberries contained only 0.04 mg/kg and 0.2 mg/kg benzaldehyde, respectively (Honkanen et al., 1980; Hirvi and Honkanen, 1983). Mazza and

Hodgins (1985) also reported that homogenized saskatoon berries, distillates, and benzaldehyde had similar warm, sweet, almondy odours.

Researchers have shown that grinding and heating affected glycosidically bound benzaldehyde (Schmid and Grosche, 1986). Thus, if the benzaldehyde found in saskatoon berries by Mazza and Hodgins (1985) occurred from amygdalin breakdown their results may be partially explained by :

1) a lack of seeds in the juiced fruit giving a lower juice GC benzaldehyde peak area than that for whole fruit;
2) enzymatic release of benzaldehyde following the physical breakdown of fruit and heat treatment; and
3) enzymatic production of benzaldehyde during the grinding and heating of fruit for AOAC method analysis.

Analytical techniques which minimize creation of artefacts must be carried out to determine if benzaldehyde is the major flavour volatile of fresh saskatoon berries.

1.4 Analytical Methodologies for Flavour Constituents

In the 1970's, studies were conducted on non-volatile and volatile constituents of saskatoon berries using available methodologies and equipment. Improvements in high pressure liquid chromatography (HPLC) and GC/MS instrumentation and new methods have increased the sensitivity and reproducibility of these analyses. Studies on sugar, acid, and flavour components of saskatoon juice using new instrumentation should provide further information on the types and quantities of these non-volatile and volatile constituents which significantly impact the quality of saskatoon juice.

1.4.1. Methodologies for Non-Volatile Constituents

Wolfe and Wood (1971) using a 1956 colorimetric method identified fructose, glucose, and sucrose in saskatoon berries but were concerned about the reliability of the sucrose data. Currently, an HPLC method using a specialized carbohydrate column, a water/acetonitrile mobile phase, and a refractive index detector is the most widely used technique for quantifying sugars in plant extracts (Shaw, 1988). AOAC method 31.145-31.149 (AOAC, 1990) is consistent with the above approach and has been used successfully by the Food Laboratory Services Branch, Alberta Agriculture, to analyse sugars in saskatoon berries (Field, 1991). Analysis of sugars in saskatoon berry juices via this method may overcome earlier unreliable sucrose data (Wolfe and Wood, 1971).

Wolfe and Wood (1971), employed an ion exchange technique and found malic, oxalic, and citric acids in unripened saskatoon berries and only malic acid in ripe fruit. Panther and Wolfe (1972) using both titrimetric methods and polarographic assays, did not determine ascorbic acid in saskatoon juices. Researchers have found malic, citric, quinic, and ascorbic acids in other rosaceous fruits (Coppola and Starr, 1986).

Current methods of organic acid analysis include HPLC ion-exchange or ion-pairing to separate ionised or ionisable compounds. Reverse phase (non-polar stationary phase and polar mobile phase) methods with bonded columns are favoured as they are rapid, reproducible and allow separation of compounds with wide ranges of polarity (Simpson, 1982). The most commonly used columns are C_{18} alkyl groups bonded to a surface silica matrix.

Accurate quantitation and resolution of early eluting organic acids, such as quinic acid, in fruits are difficult. Coppola and Starr (1986) successfully used a reverse phase system, and one or two LC-18 columns in tandem with a phosphate buffer and UV detection at 214 nm, to separate quinic, malic, and citric acids from apple juice and cranberry juice cocktail. Use of the second column improved quinic acid resolution. Ascorbic acid was also determined by this method although it coeluted with some shikimic acid. Since it has been successful for organic acid analysis in related fruits, this method (Coppola and Starr, 1986) would appear to be suitable for determination of organic acids in saskatoon berry juices.

Reverse phase ion-pairing techniques rely on extraction of an ionised water soluble compound into an organic solvent by a counter ion. The ion pair behaves like a nonionic polar molecule in the organic solvent and is readily separated on reverse phase C-18 columns with the elution of more polar compounds first. This technique, using tetrabutylammonium at pH 7.4 as the ion-pairing reagent, has been employed to separate organic acids (Simpson, 1982). Although published reports of its use for fruit juices are lacking, reverse phase ion pairing may have potential for determination of organic acids in saskatoon juices.

1.4.2. Methodologies for Volatile Constituents

Volatile aroma constituents of fruit flavours have been determined using headspace, distillation, or solvent extraction techniques to extract the volatiles and capillary GC/MS to identify and quantify the volatiles. Headspace analysis methods collect low boiling volatiles and concentrate them on some form of trap before desorption into a GC column. Hirvi and Honkanen (1982) showed that many of the character impact components for strawberries were too volatile to be determined by this method. Atmospheric or vacuum distillation is frequently employed to isolate essential oils from spices. Distillation has not been widely used for isolation of berry volatiles as many of the compounds may be thermally degraded (Honkanen and Hirvi, 1990). Solvent extraction is the most widely reported isolation technique for analysis of berry volatiles. Diethyl ether, the commonly used solvent, gives high recoveries and evaporates readily during sample concentration. Comparing headspace, distillation, and solvent extraction techniques for flavour isolation from an aqueous model system, Leahy and Reineccius (1984) concluded that solvent extraction was the most efficient method. Two constraints of this methodology noted were its limitation for use with fat-free foods and its potential loss of very low boiling volatiles (Leahy and Reineccius, 1984).

Previous studies on saskatoon berry volatiles have used headspace and distillation techniques (Mazza, 1980; Mazza and Hodgins, 1985) which may have led to the formation of artefacts due to enzyme activity and/or heat degradation. A solvent extraction technique, which minimizes the application of heat, may overcome these problems and provide a more accurate profile of the flavour volatiles of the fresh saskatoon berry. Earlier workers (Mazza, 1980; Mazza and Hodgins, 1985) found only a few GC peaks and identified only one, benzaldehyde. The use of solvent extraction, GC, and MS should permit identification of the volatile flavour constituents which contribute to the unique aroma of saskatoon juice.

1.5 Sensory Methodologies

1.5.1. Quantitative Descriptive Analysis

Sensory evaluation utilizes humans and their senses as instruments to evaluate the quality attributes of food products. Quantitative descriptive analysis (QDA) (Stone et al., 1974) is a technique which describes and quantifies sensory differences among products. The method uses all the senses to describe and measure characteristics such as appearance, taste, and texture of products. QDA has been employed to describe and quantify differences among fruits such as grapes (Francis et al., 1992), apples (Williams and Carter, 1977), and canned peaches (Spencer et al., 1978).

To provide reproducible meaningful information a QDA panel must be trained to understand the vocabulary and to use it to consistently quantify their measurements. Detailed procedures for training QDA panels have been documented by ASTM (1985) and Stone and Sidel (1985). The approach includes: recruitment of 24 - 36 panelists, group development of sensory descriptors with standards, selection of panelists by a series of triangle tests that determine panelists' ability to differentiate among samples with varying amounts of sensory characteristics, development and use of 15 cm anchored descriptive line scales, and final selection of panelists based on their ability to give accurate reproducible data among test products. The 6 - 12 member panel functions as a calibrated instrument capable of accurately reproducing specific sensory measurements.

Training of a QDA panel requires considerable time and effort. Panel training is necessary to overcome initial variations among the panelists due to differing:

1) vocabulary used to describe the same stimulus;
2) scores for the same quantity of a given characteristic;
3) ranges in a given attribute's scores when differentiating among products tested; and
4) responses among sessions (Dijksterhuis and Punter, 1993).

Although a QDA panel takes time to "calibrate" it has significant advantages for food research. The first advantage of panelists lies in the sensitivity of the human nose. The nose is more sensitive to some compounds than analytical techniques and responds differently to volatile compounds than a GC. A small GC peak may be more important to human perception of odour and flavour than a larger peak. The GC records a linear response to the concentration of a volatile whereas human olfactory systems register the change as a power function (Wick, 1965). Secondly, panelists' perception is based on the overall effect of all chemical constituents in a product. Sugars and acids in fruit may be measured individually but the sensory perception of sweetness or sourness associated with these constituents is dependent on the presence of both components. Ultimately, it is the human response which best evaluates flavour quality.

The QDA methodology relies on analysis of variance for data analysis and interpretion. Each characteristic is analysed as treatment by subject for a number of replicates. Panel replications provide an indication of panel reliability.

Published literature on sensory descriptors of saskatoon berry flavour is limited to a reference by Mazza and Hodgins (1985) that homogenized berries smelled warm, sweet, and almondy. A complete sensory profile of saskatoon berries, obtained with a QDA panel, would provide information that is essential to understand and interpret current and future instrumental data.

1.5.2. Free-Choice Profiling

Free-choice profiling (FCP) is a sensory technique which describes and quantifies sensory differences among products (Williams and Langron, 1984). The major difference between QDA and FCP is that QDA uses trained panelists while FCP uses untrained judges.

Free-choice profiling, originally developed by the Agricultural and Food Research Council (Williams and Langron, 1984), involves: familiarizing untrained consumers with the products to be evaluated and having each panelist develop a list of sensory descriptors for product attributes as well as anchor terms for the scale ends; preparing individualized scorecards; and presenting panelists with samples of test products for quantitative assessment. Care must be taken to ensure that the consumer's scales are unidirectional and not hedonic. Hedonic question(s) may be included at the end of the free-choice profile scorecard.

Since FCP is a relatively new technique, published methods differ. A variety of panel selection, vocabulary development, scoring, sample presentation, and comparison techniques have been reported (Guy et al., 1983; Williams and Langron, 1984; Steenkamp and van Trijp, 1987; McEwan et al., 1989; Beilken et al., 1990; Beilken et al., 1991; Lynch et al., 1991; ;).

The process of selecting panelists and the size of the panels have varied widely. The Agricultural and Food Research Council used FCP to evaluate eight ports (Williams and Langron, 1984). The ten "untrained" panelists were buyers, producers, experienced conventional profilers, and scientists all involved with port. Other researchers have utilized FCP with small groups of panelists, previously trained in conventional profiling techniques, for: coffee, n=10 (Williams and Langron, 1984); meat patties, n=12 (Beilken et al., 1991); cheese, n=8 (McEwan et al., 1989); processed meats, n=12 (Beilken et al., 1990) and irradiated turkey, n=10 (Lynch et al., 1991). Except for Lynch et al. (1991) who used one replication, other workers have employed three or four replicates. Two studies utilized large groups of naive consumers to evaluate whisky, n=93 (Guy et al., 1983) and meat, n=64 (Steenkamp and van Trijp, 1987). Thomson (1989) indicated that FCP has potential for use with naive consumers, provided they are able to adequately verbalize the differences they perceive among products, but noted the need for further studies.

Development of a vocabulary of descriptors for each panelist has varied depending on the panel selection. Researchers (Williams and Langron, 1984; McEwan et al., 1989; Beilken et al., 1990; Beilken et al., 1991; Lynch et al., 1991) using 8-12 experienced panelists provided samples for evaluation during vocabulary development sessions. One to five sessions were held for individual vocabulary and scorecard development. In a shelf-life study of meat, Lynch et al. (1991) permitted experienced panelists to add vocabulary words as new aroma notes developed. With untrained consumers, Thomson (1989), Steenkamp and van Trijp (1987) and Guy et al (1983) employed a variety of techniques for FCP vocabulary development. Thomson (1989) utilized a triad system whereby three products were presented to panelists who were requested to explain why 'product a' and 'product b' were similar and why they differed from 'product c'. Various product combinations were evaluated until an adequate vocabulary evolved. Steenkamp and van Trijp (1987) required panelists to place a variety of products into two groups and explain why. Product grouping continued until a complete vocabulary was established. Guy et al. (1983) provided instruction on the FCP method and permitted consumers to generate their own vocabulary based on eight samples provided during one session.

Two types of systems have been used for scoring FCP descriptors. Williams and Langron (1984) originally used a six point intensity scale (0=absent --> 5=very intense) for ports and coffee. Steenkamp and van Trijp (1987) employed a similar five point scale. Others (Beilken et al., 1991; McEwan et al., 1989; Beilken et al., 1990; Lynch et al., 1991) have used 10 cm line scales for scoring attributes.

Experimental conditions and sample presentation for FCP depended largely on panel size. Studies (Williams and Arnold, 1985; McEwan et al., 1989; Beilken et al., 1990; Beilken et al., 1991; Lynch et al., 1991) involving small panels were conducted in sensory evaluation booth settings. Consumer panels (n>50) were conducted under laboratory conditions (Steenkamp and van Trijp, 1987) or in-home (Guy et al., 1983).

FCP procedures have been compared with conventional profiling techniques and/or evaluations of product acceptability. Several workers (McEwan et al., 1989; Beilken et al., 1990; Beilken et al.,1991) attempted to relate FCP to consensus profiling. The free-choice experiment was conducted first and then the panelists developed a common vocabulary for consensus profiling before repeating the experiment. Both panels produced similar results but consensus profiling differentiated products to a greater degree than FCP likely due to the common vocabulary and panel training (Beilken et al., 1990; Beilken et al.,1991; McEwan et al., 1989). To relate product acceptability to sensory attributes, hedonic data from 50-116 consumers were correlated to FCP results by preference mapping techniques (McEwan et al., 1989; Beilken et al., 1990; Beilken et al.,1991). Only Guy et al. (1983) compared data from a conventionally trained profiling panel and those of a large consumer FCP panel. Both panels determined similar three dimensional configurations to express differences among eight whiskeys. In both configurations, axes one and two were associated with similar descriptors but the third axes were not related to common descriptors.

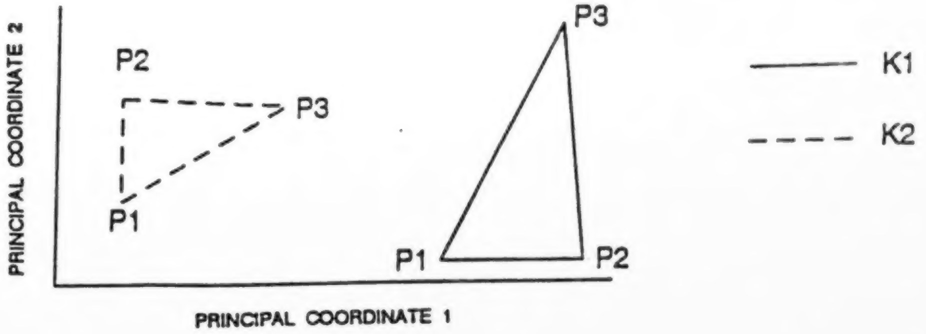
Many research studies have used QDA to provide detailed sensory information for quantification of product differences. Only a few studies have been published using FCP. Use of a trained QDA panel and an untrained consumer FCP with hedonic responses could be advantageous in determining differences among saskatoon juices due to processing techniques. The trained panel will provide sensory parameters of saskatoon juices which may be correlated with instrumental data. The frequency of descriptors used by a FCP with a large consumer group (n>50) may indicate which characteristics of saskatoon juice are most significant. Although a consumer panel is generally less discriminating than a trained panel, consumer data may help relate the trained panel results to the marketplace. Hedonic responses obtained from consumer FCP may assist in determining the effects of any differences on product acceptability.

1.5.3. Generalised Procrustes Analysis

Generalised Procrustes analysis (GPA) is a statistical technique used to interpret differences among products based on data from each of the individuals in a group rather than through interpretations based on the means of data from a group (Gower, 1975). GPA is required for FCP as each panelist creates a unique data matrix. Each individual (K) provides information on products (P) according to unique descriptors (D). Data matrices vary according to: 1) the scores given for similar characteristics, eg. K1 may give values ranging from 1-4 for sweetness of all the products while K2 may assign values of 10-14 for sweetness; 2) the range in scores used to differentiate among products, eg. K1 may give scores of 5-9 for the different products, whereas K2 may show greater differences in product scores ranging from 3-11;

and 3) the number and order of descriptors, eg. K1's first descriptor D1 may be a colour descriptor whereas K2's first descriptor D1 may be for sweetness.

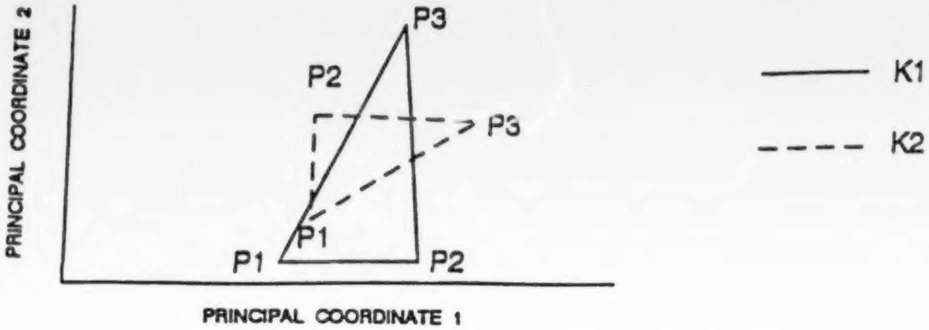
GPA adjusts the data matrices to overcome the variations noted above through : 1) translation; 2) scaling; and 3) rotation. Given two individuals, K1 and K2, using two descriptors each, D1 and D2, to assess differences among three products, P1, P2, and P3, the resulting configuration can be plotted two dimensionally as shown in Figure 1.7.



[Adapted from McEwan and Hallett (1990)]

Figure 1.7. Pre-Procrustes data configurations.

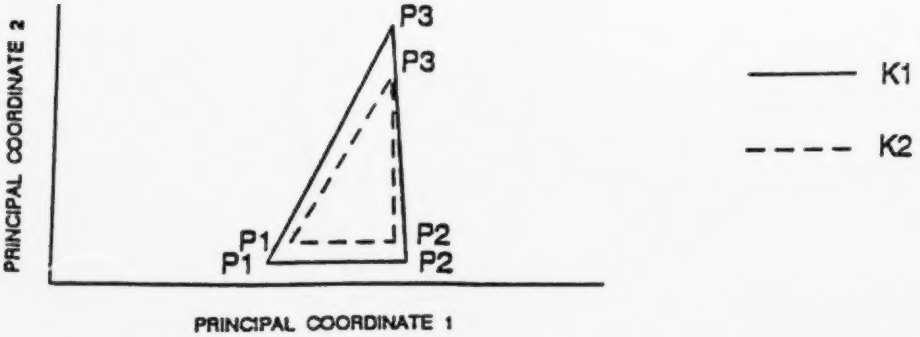
The first step in GPA is translation of each configuration to a common centroid (Figure 1.8). Translation removes the individual main effect and thus adjusts for each individual's use of a different portion of the scales.


P3
P2
P3
P1
P1
P2
K1
K2
PRINCIPAL COORDINATE 1
PRINCIPAL COORDINATE 2

[Adapted from McEwan and Hallett (1990)]

Figure 1.8. GPA translation of data configurations.

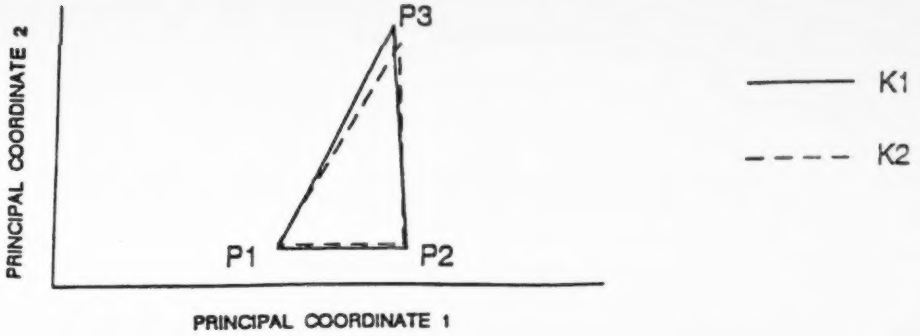
Each configuration can then be rotated until the shapes align (Figure 1.9). Rotation aligns attributes so they are located close to each other on the plot.


P3
P3
P1
P1
P2
P2
K1
K2
PRINCIPAL COORDINATE 1
PRINCIPAL COORDINATE 2

[Adapted from McEwan and Hallett (1990)]

Figure 1.9. GPA rotation of data configurations.

Subsequently the data are scaled to adjust for individual variations in the magnitude of differences judges have indicated among the samples. The effect on the configurations is shown in Figure 1.10.



[Adapted from McEwan and Hallett (1990)]

Figure 1.10. GPA scaling of data configurations.

In the preceding example K1's two descriptor's D1 and D2 corresponded to principal coordinates 1 and 2, respectively. K2's descriptors D1 and D2 originally corresponded to principal coordinates 1 and 2. GPA rotated the positions of K2's descriptors so that in the end D1 corresponds to principal coordinate 2 and D2 corresponds to principal coordinate 1.

The structure of each original configuration is preserved since the relative positions of the three products are still the same as they were in Figure 1.7. The final consensus configuration (Figure 1.10) is used in the interpretation of each individual's data.

Free-choice panelists use varying numbers of D descriptors for a given product. GPA handles variations in the D dimension of the data matrices by inserting zeros. The number of principal coordinates which can be analysed is limited by the lowest number of D descriptors used by one judge. In many cases each principal coordinate is composed of more than one descriptor. In the example above, product P3 may have differed from both P1 and P2 in sweetness and darker colour. Principal coordinate one would thus represent both sweetness and dark colour. Publications use the terms principal coordinate, principal axis, or dimension interchangably (Arnold and Williams, 1986)

For analysis of values of more than two panelists (K) and descriptors (D), GPA goes through a series of iterative steps (Oreskovich et al., 1991). Each individual configuration is translated, rotated, and scaled until the sum of the squares of the distances between the individual configurations and a target configuration is minimized. A mean of all individual configurations is determined which becomes the new target configuration and the process repeats itself until a predetermined tolerance is reached. The final mean configuration of all individual configurations is known as the consensus configuration or consensusspace.

GPA has also been used to analyse conventional QDA data and to obtain information on individual panelist's performance (Williams et al., 1981; Dijksterhuis and Gower, 1993). Because the analysis is conducted on each individual's data it is possible to determine if there are problems with : 1) panelists using different levels of the scale for scoring via translation of the data; 2) panelists using different ranges to express differences among samples via scaling the data; and 3) panelists not using the same terminology to describe a stimulus via data rotation. GPA may be used throughout training to focus on individual panelists needs and/or to select the final group of panelists.

The use of GPA to analyse sensory data was first reported by Gower (1975). More recent publications frequently use computerized versions of GPA including: GENSTAT, SAS, and OP&P's Procrustes-PC versions. These programmes provide statistical interpretation of the variations among the products and panelists. A major difference among the programmes is the use of variation scaling by the Procrustes-PC version. This programme reports analysis of variance tables which are expressed as percentages of the total amount of variation. As Procrustes-PC was available for this research the following discussion will focus on this programme.

The total variation (V_T) is the sum-of-squares over all K individual sets, N products and D_K descriptors :

$$V_T = \sum_k^K \sum_i^N \sum_j^{D_k} X_{kij}^2$$

[Adapted from Dijksterhuis and van Buuren (1989)]

V_T can be partitioned in four parts (V_{IN}, V_{OUT}, V_W, and V_P) according to products, panelists, and principal coordinates. The first two parts, V_{IN} and V_{OUT}, are the variations explained by the number of principal coordinates of the consensus configuration and the higher principal coordinates of the consensus configuration, respectively. They are usually reported together as V_C (C=consensus). The other two parts, V_W and V_P, are residuals arising from the variation lost in averaging the individual spaces to the consensus configuration (V_W) and the variation lost in the process of projecting data onto the subspaces (V_P).

Analysis of variance tables are produced for products, panelists, and principal coordinates. The products table indicates V_C and V_W to show the amount of variation each product contributes to the consensus (V_C) and how much variation there is regarding each product's final positioning (V_W), respectively. The panelists table indicates V_W for each judge. A large V_W value indicates a judge who does not agree with the consensus configuration. The principal coordinate table indicates V_C and V_W for each principal coordinate. Variation attributable to V_C signifies the relative importance of that principal coordinate in explaining the results. If an analysis for two principal coordinates is requested and less than 50% of the variation is explained by the model another analysis, using three or more principal coordinates, may be required to better understand the results.

1.5.4. GPA Correlation of Sensory and Instrumental Data

Data from sensory and instrumental research are frequently correlated (Paule and Powers, 1989; Spencer et al., 1978). Publications to date illustrate use of GPA for individual sensory or instrumental data sets which describe differences among products. Theoretically, it should be possible to analyse both sensory and instrumental data together by GPA.

Liardon et al. (1984) compared GPA analyses of QDA sensory results and GC analytical data for fifteen coffees. The authors found that, when superimposed, each of the resulting sensory and instrumental configurations was similar but none of the sensory characteristics was related to a specific GC component. They concluded that subjective coffee quality parameters were linked to a combination of volatile components. Other published reports of the combined analysis of sensory and instrumental data sets using GPA are lacking. Incorporation of sensory and instrumental data in one GPA may enhance the determination of relationships among the data. Moreover, combining GPA of sensory data with GC data, prior to volatile compound identification may assist in predicting which constituents have a significant impact on specific sensory characteristics and thus focus identification efforts.

This study on processed saskatoon juices is expected to provide detailed information on sensory characteristics and volatile constituents. A combined GPA of the sensory and instrumental data may assist in focussing volatile constituent identification and interpreting correlations among sensory and instrumental data.

1.6. Objectives of the Thesis

In summary, the harvest of saskatoon berries in Alberta is sufficient to warrant the development of value-added processed products, but information on commercial processing methods and the chemical, physical, and sensory characteristics related to saskatoon berries is lacking. This information is required to facilitate the development of high quality saskatoon products.

This thesis focuses on the production of saskatoon juice since juice processing is the initial step in the manufacture of a variety of consumer products. The objectives of the thesis are:

1) to determine the chemical constituents of saskatoon juice related to flavour (chapters two and three);
2) to determine the effect of cold, hot, and enzyme processing methods on instrumental and chemical constituents (chapters two, three and four) and flavour (chapters five and six);
3) to define the sensory characteristics of saskatoon juice using trained QDA (chapter five) and consumer FCP (chapter six) panels;
4) to compare FCP and QDA sensory methodologies using GPA (chapter six); and

5) to establish relationships among instrumental and sensory data using GPA (chapter seven).

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Chapter 2 Effects of Hot and Cold Processing Methods on Chemical Flavour Constituents of Processed Saskatoon Berry Juice[1]

2.1. Introduction

Saskatoon shrubs, *Amelanchier alnifolia* cv. Smoky, have been grown commercially in Alberta since 1972 (Davidson, 1986). Alberta's saskatoon berry yields are expected to reach 500,000 kg by the late 1990's as bushes mature and more acreages are planted (Hausher, 1988). This quantity of fruit will provide a basis for development of the value-added processed saskatoon berry industry.

Currently saskatoon wine, jellies, and syrups are the most prevalent processed saskatoon products in the domestic marketplace. These products require that saskatoon berries be juiced prior to processing into their final forms. Mazza et al. (1978) found juicing cold saskatoon berries gave poor yields. Hot juicing of blueberries (Yang, 1984) and cherries (Tressler et al., 1980) gave greater yields than cold juicing. A comparison of the effects of cold and hot juicing of saskatoon berries on yield has not been published. Yield optimization during the juicing process is important to minimize the production costs of saskatoon wines, jellies, and syrups.

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[1] An abstract of this section has been published. Lutz, S.E., Dowdell, L.R.J., Hawrysh, Z.J. and Sporns, P. 1991 Can. Inst. Food Sci. Technol. J. 5:190/AB

Mazza (1979, 1980) found juicing saskatoon berries significantly reduced (45.4%) total headspace volatiles as compared to whole berries and that the resulting jelly had a weak flavour. Information on the flavour constituents in saskatoon berries is required to understand the effects of processing on resultant juice flavour. Research on saskatoon berry aroma identification has been limited to one study (Mazza and Hodgins, 1985) which used a distillation technique to identify benzaldehyde as a major constituent. Broderick (1975) reported that benzaldehyde in cherries was derived from enzymatic hydrolysis of amygdalin, a cyanoglucoside, in the stone. Majak et al. (1981) found amygdalin glucoside in saskatoon foliage and fruit. Benzaldehyde, determined by Mazza and Hodgins (1985) in saskatoon berries, may have been produced from the seeds as a result of the analytical methods used. Research on the identification of aroma constituents, using other instrumental techniques, is necessary to understand and optimize the overall flavour of saskatoon juice.

The present study was conducted to identify the volatile constituents in cold and hot processed saskatoon juices by gas chromatography/mass spectroscopy (GC/MS), and to determine the effect of these processing methods on juice yield.

2.2. Materials and Methods

A total of ninety-six kg of individually quick frozen (IQF) saskatoon berries, from the Peace Country Fruit Producers Cooperative at Hythe, AB, was randomly assigned in 12 kg lots to either a cold or hot processing treatment. Each treatment was replicated four times. Berries allotted to cold processing were juiced, at 3°C, with a Brown screw type pulper finisher (model 3600 / 0.60 screen, Brown International Corp., Covina, CA); those assigned to hot processing were heated to 77°C in a steam jacketed kettle (Groen 5 US gallon, Groen Process Equipment Group, Elk Grove Village, IL) and held for 5 min before juicing in the Brown pulper finisher. Processing of the two juice treatments was randomized. Juice from each of the treatments was weighed to determine the percentage yield (wt. juice / wt. berries X 100%). Juice soluble solids were determined by refractometry (Reichard Abbe Mark II, AO Scientific Instruments, Buffalo, NY) at 20°C and the pH with a pH meter (Fisher Accumet Model 750 with Orion Ross #8165BN electrode, Fisher Scientific Co., Pittsburgh, PA). Soluble solids and pH of each processed juice were measured in duplicate and the average was recorded. For GC analyses, 2,6-dimethyl phenol (5ppm) was added to the processed saskatoon juices as an internal standard. Juices were packed in 250 mL glass jars, sealed with polyvinyl-lined metal lids, and frozen (-18°C) in a blast freezer for subsequent analyses.

Thawed processed saskatoon juices were solvent extracted according to the method of Leahy and Reineccius (1984). For each juice treatment within a replicate, 500 mL juice was thawed in a 20°C water bath for approximately 30 minutes. Then, 400 g of saskatoon juice at 20°C and 50 g redistilled diethyl ether were combined in a 500 mL separatory funnel and shaken for 3 min. After 40 min, the solvent in the separatory funnel was decanted and centrifuged (Damon CRU-5000 centrifuge, International Equipment Co., Needham, MA) at 1,500 rpm for 10 min. Solvent from the top of the centrifuge bottle was decanted and collected in a 250 mL separatory funnel. Juice remaining in the centrifuge bottle was recombined with the juice in the 500 mL separatory funnel and with 50 g redistilled diethyl ether and successively separated two more times. Combined solvent extracts were dried by pouring over dried anhydrous sodium sulfate and evaporated to approximately 20 mL on a Calab rotovap (Model 5120, California Laboratory Equipment Co., St. Emeryville, CA) at -55 kPa. Concentrated saskatoon juice solvent extracts were stored (-23°C) in 125 mL test tubes. Preliminary trials indicated >95% internal standard recovery using this method. Solvent extraction was repeated four times for each juice treatment within a replication.

Saskatoon juice solvent extracts were analysed, based on the method of Kallio et al. (1984), using a Hewlett Packard 5840 A GC / 5987 MS system (Hewlett Packard Co., Corvallis, OR) fitted with a slightly polar capillary column (Econo-cap SE 54, 30 m X 0.25 mm id X 0.25 μm immobilized 5% diphenyl

95% dimethyl polysiloxane film, Alltech, Deerfield, IL). Helium, at a flow rate of 2 mL/min, was used as the carrier gas. Separation conditions included an initial oven temperature of 50°C for 2 min and a linear programme of 10°C/min to 275°C. A 2μL sample was injected without splitting into an injection port at 220°C. Chromatogram peak areas were recorded as a percentage of the 2,6-dimethyl phenol standard. Preliminary GC trials of duplicate samples showed good reproducibility with 1-11% differences between major peak areas. The GC/MS interface and ion source temperatures were 275°C and 200°C, respectively. Mass spectra were obtained with an ionization energy of 70 eV and within the mass range of m/z 40-350. A Hewlett Packard NIH/EPA/MSDC mass spectral database #041 was used for tentative chemical identification of chromatogram peaks. For comparison of cold and hot processed juices, a total of thirteen peaks were selected based on replicate reproducibility ($\pm$ 50% of total peak area) and a peak area >1.00% of the internal standard peak area.

Data for yield, soluble solids, pH and areas of thirteen peaks from the processed saskatoon juice extracts were subjected to analysis of variance. Sources of variation were : processing treatments (t=2), replications (r=4), and for instrumental tests (i=4).

2.3. Results and Discussion

Mean yields, soluble solids, and pH for cold and hot processed saskatoon juices are presented in Table 2.1. Hot processed juice yield was significantly higher ($p \leq 0.05$) than that of cold juice. The yield of cold processed juice ranged from 36-41%; hot processed juice yield ranged from 43-52%. These findings are consistent with studies on blueberries (Yang, 1984) and cherries (Tressler et al., 1980) which reported greater yields with hot processed than cold processed juices. The soluble solids content of hot processed juice was significantly higher ($p \leq 0.05$) than cold processed juice. The soluble solids contents of cold processed juices ranged from 15.7% to 16.6% while hot processed juices were all 16.7%. The pH of cold and hot processed saskatoon juices did not differ. For several varieties of saskatoon berries, Green and Mazza (1986) reported that soluble solids content ranged from 12.8% to 16.9% and the pH from 3.8 to 4.5. Thus, soluble solids content and pH data for saskatoon juices in the present study are in agreement with earlier results (Green and Mazza, 1986).

Table 2.1. Means[1] for percentage yield, percentage soluble solids, and pH of processed saskatoon berry juices.

	Processed Juice Treatment		
Variable	Cold (3°C)	Hot (77°C)	SEM[2]
% Yield	38.3	47.0	1.54*
% Soluble Solids	16.0	16.7	0.14*
pH	3.9	3.9	0.00

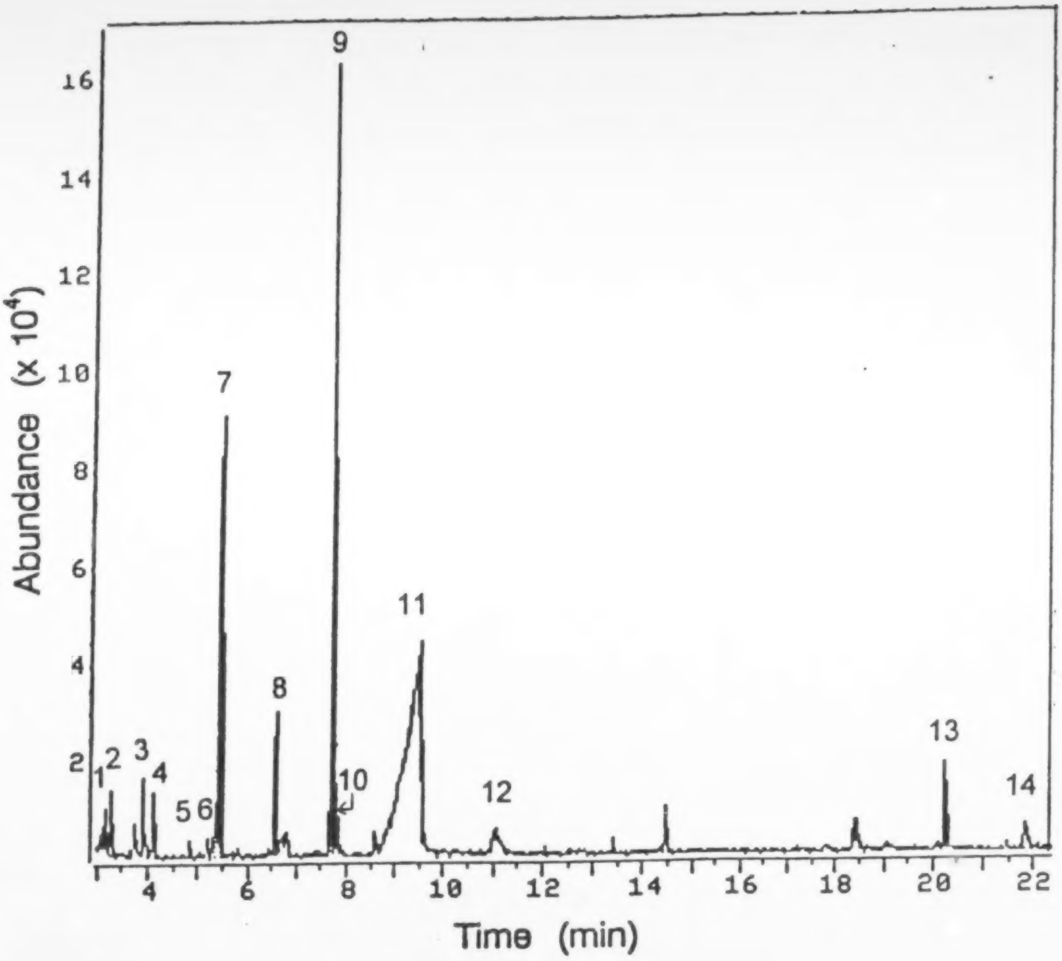
[1] Means are the average of 4 determinations

[2] Standard error of the means

* Means are significantly different at $p \leq 0.05$

Typical GC profiles of volatile constituents in solvent extracts of cold and hot processed juices are presented in Figures 2.1 and 2.2, respectively. Mean peak areas for fourteen GC peaks for the two treatments are presented in Table 2.2.

Peaks 1-4 and 6, unidentified volatiles of low (<100) molecular weight, were found in significantly ($p \leq 0.05$) higher quantities in cold than hot processed saskatoon juice extracts. Perhaps heat during hot processing caused evaporation of some of these low molecular weight, low boiling point compounds. These low molecular weight compounds may have an important effect on the flavour of hot processed saskatoon juice. The significant reduction in low molecular weight volatiles in hot versus cold processed saskatoon juices in the present study may account for the weak flavour noted by Mazza (1980). Peaks 7,8, and 10-12 have been tentatively identified as benzaldehyde ($p=0.88$), benzyl alcohol ($p=0.91$), phenyl ethanol ($p=0.96$), benzoic acid ($p=0.85$), and mandelonitrile ($p=0.85$). Hot processed juice contained significantly ($p \leq 0.01$) higher quantities of benzaldehyde, benzyl alcohol, and phenyl ethanol than cold processed saskatoon juice. In contrast, the relative concentrations of benzoic acid and mandelonitrile in both processed saskatoon juices were similar. In crushed cherries mandelonitrile was produced from amygdalin via glucosidase hydrolysis (Broderick, 1975). Subsequent action of hydroxynitrile lyase on mandelonitrile resulted in benzaldehyde and hydrocyanic acid (Wong, 1989). Amygdalin, a



1.-6. unknown

7. benzaldehyde (T)

8. benzyl alcohol (T)

9. 2,6 di-methyl phenol (std)

10. phenyl ethanol (T)

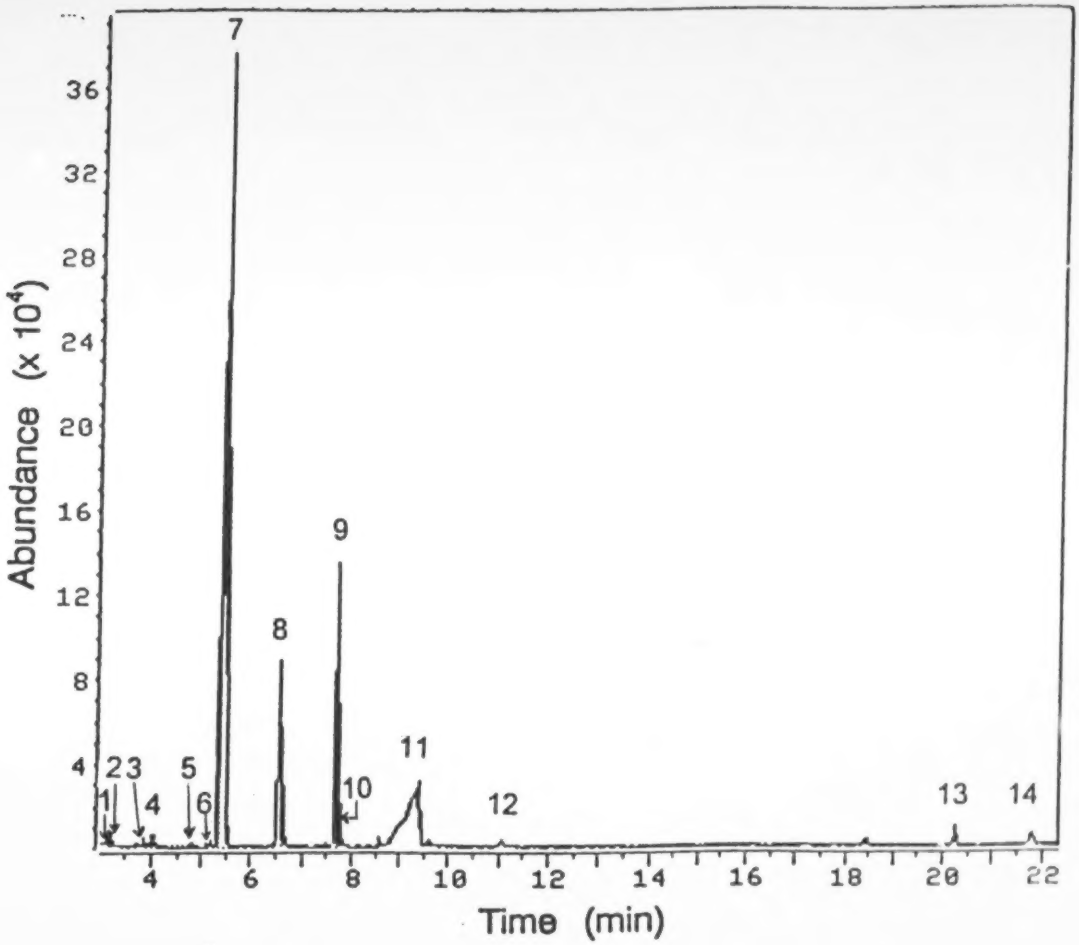
11. benzoic acid (T)

12. mandelonitrile (T)

13.-14. unknown

(T) = tentative identification

Figure 2.1. GC profile of volatile constituents in cold (3°C) processed saskatoon juice, using a SE54 column. Peak numbers correspond to Table 2.2.



1.-6. unknown

7. benzaldehyde (T)

8. benzyl alcohol (T)

9. 2,6 di-methyl phenol (std)

10. phenyl ethanol (T)

11. benzoic acid (T)

12. mandelonitrile (T)

13.-14. unknown

(T) = tentative identification

Figure 2.2. GC profile of volatile constituents in hot (77°C) processed saskatoon juice, using a SE54 column. Peak numbers correspond to Table 2.2.

Table 2.2. Means[1]and standard errors for gas chromatographic analysis (SE54 column) of volatiles in processed saskatoon berry juices.

Peak #	Name[2]	Retention Time (min)	Processed Juice Treatment		SEM[3]
			Cold (3°C)	Hot (77°C)	
1	unknown	3.16	3.47	2.42	0.20*
2	unknown	3.23	5.70	2.80	0.44**
3	unknown	3.80	50.60	32.00	4.60*
4	unknown	4.08	6.30	4.00	0.35**
5	unknown	4.83	1.90	2.00	0.20^{ns}
6	unknown	5.20	3.10	2.20	0.18*
7	benzaldehyde	5.41	61.30	682.40	15.37**
8	benzyl alcohol	6.57	15.20	89.30	2.25**
10	phenyl ethanol	7.78	5.50	15.30	0.77**
11	benzoic acid	8.55	39.50	36.40	3.31^{ns}
12	mandelonitrile	11.10	18.50	25.70	5.12^{ns}
13	unknown	20.20	7.40	4.70	1.02^{ns}
14	unknown	21.80	8.40	4.60	1.00*

[1] Means are the average of 16 determinations (4 per 4 replicates) expressed as percentage of internal standard

[2] Tentative identification

[3] Standard error of the mean

ns Not significant

*, ** Significantly different at $p \leq 0.05$ and $p \leq 0.01$, respectively

cyanoglucoside, has been determined in saskatoon foliage and fruit (Majak et al., 1981). Tentative identification of mandelonitrile and benzaldehyde in the chromatograms of cold and hot processed saskatoon juices indicates that these enzymatic reactions may have occurred. Salles et al. (1988) determined that benzyl alcohol and phenyl ethanol were released through the enzymatic hydrolysis of glycosidically bound volatiles. Hot processing of apricots (Chariote et al., 1981) and cherries (Schmid and Grosche, 1986) increased release of glycosidically bound volatiles including aromatic alcohols. Thus, in the present research, the large amounts of benzaldehyde, benzyl alcohol, and phenyl ethanol in hot versus cold processed saskatoon juice may indicate faster enzymatic hydrolysis of amygdalin in the presence of heat.

Compared to cold juice, benzaldehyde, benzyl alcohol, and phenyl ethanol increased eleven, seven, and three fold, respectively, in hot juice. These compounds are known to contribute distinctive aromas: benzaldehyde a characteristic sweet almondy aroma; benzyl alcohol, a medicinal note; and phenyl ethanol, sweet honey and floral/ rose aromas (Dravnieks, 1985; Stevens et al., 1970). Thus, the combination of these three aromatic components and their concentration in hot processed juice extracts may be important to the unique aroma of the finished product.

Mass spectra of the two remaining (rt>20 min) unidentified components (Table 2.2) indicate that they are high molecular weight (>250) compounds, possibly forms of benzene-dicarboxylic acids (p=0.75). Peak 13 was present in similar concentration in both juices; however, peak 14 was detected in significantly ($p \leq 0.05$) lower quantitites in hot than cold processed juice. Heat and/or enzyme reactions may account for the decrease in peak 14 in hot processed saskatoon juice. These high molecular weight compounds are probably not of major importance to the overall aroma of saskatoon juices.

Findings of this study indicate that the volatile constituents in hot processed saskatoon juice differed from those of cold processed juice. Hot processed juice extracts contained significantly higher quantities of benzaldehyde, benzyl alcohol, and phenyl ethanol than cold processed saskatoon juice extracts. These three aromatic constituents in hot processed juice may create a different flavour profile than that of cold processed juice. There were significantly lower amounts of unidentified low molecular weight volatiles in hot versus cold processed saskatoon juice extracts. Further research is needed to determine unidentified volatile constituents in cold and hot processed saskatoon juices and their importance to the sensory characteristics of saskatoon juices.

This study also showed that hot processed saskatoon juice had higher yields than cold processed juice. This increase in yield could be economically advantageous for manufacturing of value-added saskatoon products such as wines, jellies, and syrups.

2.4. References

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Chapter 3 Effects of Four Processing Methods on Colour and Chemical Flavour Constituents of Processed Saskatoon Berry Juice

3.1. Introduction

The manufacture of consumer products from saskatoon berries frequently requires an initial juicing process. Previous research (chapter two) showed that a hot juicing process resulted in saskatoon juice with significantly higher quantities of benzaldehyde, benzyl alcohol, and benzoic acid than cold juicing. These three aromatic constituents may result from enzymatic hydrolysis in the seed (Majak et al., 1981). Other significant differences between hot and cold processed juices were also detected for a number of unidentified volatiles (chapter two).

Earlier research (chapter two) found that hot processed saskatoon berries gave higher juice yields than cold processed berries. The use of pectic enzymes and cellulases did not increase yields for saskatoon juice (Mazza et al., 1978; Mazza, 1979; Mazza, 1980) but did increase yields of blueberry juice (Yang, 1984). The laboratory hydraulic press used in the enzyme assisted saskatoon juice studies does not simulate the juicing action of commercial juicers used in the blueberry study. As juice yield is an important cost factor, further studies using commercial equipment together with enzyme and heat treatments are needed to determine the effect of enzymes on the yield of commercially produced saskatoon juice. Schreier et al. (1978) found enzyme treated apple juices contained high quantities of C_6-aldehydes and alcohols.

Research on the effect of enzyme treatments on flavour constituents of saskatoon juice is lacking.

Changes in blueberry (Yang, 1984) and cherry (Tressler et al., 1980) juice colours have been found due to oxidative enzyme reactions during cold or hot processing. Data on colour changes in saskatoon juice during processing are not available. Therefore, instrumental measurements of colour are required to provide data on the effect of processing on saskatoon juice colour.

The major sugars determined in saskatoon berries were glucose and fructose; the amount of sucrose present was too low for accurate quantification (Wolfe and Wood, 1971). Wolfe and Wood (1971) found malic and citric acids in ripe saskatoon berries; ascorbic acid was absent (Panther and Wolfe, 1972). In contrast, other rosaceous fruits contain malic, citric, quinic, and ascorbic acids (Coppola and Starr, 1986). Current analytical methods, such as HPLC ion-exchange (Coppola and Starr, 1986) or ion-pairing (Simpson, 1982), may yield further information on organic acids in saskatoon juice.

Thirteen volatile constituents were determined in saskatoon juice using a GC with a SE54 (slightly polar) column (chapter two). However, Leahy and Reineccius (1984) indicated that a GC with a carbowax (polar) column was advantageous for analysing fruit volatiles. Analysis of saskatoon juice extracts using a polar column may assist in the identification of additional volatile constituents.

The present study was conducted to determine the effect of cold, pectic enzyme, hemicellulitic and pectic enzymes, and hot processing of saskatoon berries on juice yield, colour, sugars, acids, and aroma constituents.

3.2. Materials and Methods

A total of 192 kg of blast frozen firm-ripe saskatoon berries, cv. Smoky, half from Pearson's Berry Farm, Innisfail, AB, and half from Alberta Special Crops and Horticulture Center, Brooks, AB, was thawed for 24 hrs at 3°C and randomly assigned in 12 kg lots to each of four processing treatments: cold (C), pectic enzyme (P), hemicellulitic and pectic enzyme (HP), or hot (H). Each treatment was replicated four times. A continuous process was used to juice the berries in the order of C, P, H, and HP treatments. This juicing sequence was required due to the limited amount of saskatoon berries and to minimize the transfer of chemical constituents between treatments. C processed berries were juiced at 3°C with a Brown screw type pulper finisher (Model 3600 / 0.60 screen, Brown International Corp., Covina, CA); P processed berries were tempered to 11°C, juiced by the pulper finisher, treated with 4 mL of Novo pectinex 3XL (Novo Laboratories Inc., Wilton, CT) (1 % soln.)/1000 g juice, and held in a 32°C / 38 % rH cabinet for 5 hrs; HP processed berries were heated (22°C) in a steam jacketed kettle (Groen 5 US gal, Groen Processing Equipment Group, Elk Grove Village, IL), ground in a meat grinder (Seydelmann Model D114, Maschinenfabrik Seydelmann KG, Stuttgart, Germany) at low speed with a 7mm grinder plate, held for 15 min, treated with 100 mL of Novo pectinex ultra (1% solution), held for 1 hr, and juiced by the pulper finisher; and H processed berries were heated to 77°C in a steam jacketed (Groen 5 US gallon) kettle and held for 5 min before juicing in the Brown pulper finisher. All juice treatments were centrifuged (Damon CRU-5000, International Equipment Co., Needham, MA) in 1 L bottles at 1,500 rpm for 10 min and the clarified juice was decanted. Juices were packed in 250 mL glass jars, sealed with polyvinyl-lined metal lids, and frozen (-18°C) in a blast freezer for subsequent analyses.

3.2.1. Yield

Juice from each treatment within a replicate was weighed to determine percentage yield (wt. juice / wt. berries X 100%) before and after centrifugation.

3.2.2. Colour

For colour measurements, a total of 50 mL of thawed juice was placed in a shallow 10 cm diam white dish to measure 'L', 'a', and 'b' colour values using a HunterLab tristimulus colorimeter (Model D25L-9, Hunter Associates Lab Inc., Reston, VA). Each juice treatment within a replicate was measured in duplicate.

3.2.3. Sugars

Sugar analysis in juices via high performance liquid chromatography (HPLC) was based on AOAC method #977.20 (AOAC, 1990). Ten g of thawed saskatoon juice were diluted to 100 g with distilled water and filtered (0.45 micron nylon filter paper) under vacuum. A mobile phase of 170 mL microfiltered water and 830 mL acetonitrile UV was mixed and degassed for 15 min under vacuum. HPLC parameters included a: 25μL sample injection loop, pump (Shimadzu LC-6A, Shimadzu Europa GmbH, Duisburg, Germany) operating at 1mL mobile phase/min, refractive index detector (Shimadzu R1D-6A), column heater (Waters TCM, Millipore Water Chromatography Div., Mississauga, ON) at 40°C, column (Waters CHO, 300 mm X 3.9 mm X 10 μm particle size), and an analyser (Shimadzu C-R3A). Standards for analyses were: 0.517 g D-fructose (Fisher reagent grade crystals) in 100 mL microfiltered water and 0.820 g D-glucose anhydrous (BDH reagent) in 100 mL microfiltered water. Each juice treatment within a replicate was measured in duplicate.

3.2.4. Organic Acids

HPLC organic acid analyses of juices were conducted using ion-exchange (Coppola and Starr, 1986) and ion-pairing (Simpson, 1982) techniques. Juices were thawed and diluted as per sugar analysis. A 25 μL sample was injected into an HPLC (Waters 400) fitted with a C18 column

(Phenomenex Spherisorb ODS2, 250 cm X 4.6 mm X 5 μm particle size, Phenomenex, Torrance, CA) at ambient temperature and a UV detector set at 214 nm as per Coppola and Starr (1986). A mobile phase of 27.2 g KH_2PO_4, 950 mL microfiltered water and 0.8 mL H_3PO_4 was pumped at 0.8 mL/min through the HPLC. Solutions (0.2%) of ascorbic, benzoic, citric, malic, quinic, tannic, and tartaric acids were analysed to establish standard retention times. Simpson's (1982) method was used as the basis for preparing two mobile phases: "A" = 1.7 g sodium hexane sulphonate + 25 mL methanol + 1 mL (85%) phosphoric acid + 975 mL microfiltered water; "B" = 100 g methanol. A HP 1090 HPLC was fitted with a radial compression module (Waters RCM 8X10), a radial pack cartridge (Waters 8NVC18, 100 mm X 8 mm X 4 μm particle size) and a diode array detector set at 214 nm (organic acids) and 600 nm (reference). The HPLC was programmed to pump 0.4 mL/min of 100% "A" for 1 min; then increased to 0.8 mL/min of 100% A by 5 min; then changed to 0.8 mL/min of 60% "A" + 40% "B" by 15 min; held for 5 min; and returned to 100% "A" by 30 min. Standard solutions were analysed to establish retention times and mg acid/area count unit for quantification of organic acids in diluted saskatoon juices. Ion-pairing analyses were carried out in duplicate for each experimental replicate.

3.2.5. Volatile Constituents

For GC purposes, 2,6-dimethyl phenol (5ppm) was added as an internal standard to 2 L of juice after juicing and centrifuging and prior to freezing. Thawed processed saskatoon juices were solvent extracted according to the method of Leahy and Reineccius (1984). For each extraction procedure two 250 mL jars of juice were thawed (1 hr) in a 20°C water bath. Juice was filtered with Whatman #4 filter paper under vacuum in Buchner funnels. Then 400 g of saskatoon juice at 20°C and 50 g redistilled diethyl ether were combined in a 500 mL separatory funnel and shaken for 3 min. After 40 min, the solvent in the separatory funnel was decanted and centrifuged (Damon CRU-5000 centrifuge) at 1,500 rpm for 10 min. Solvent from the top of the centrifuge bottle was decanted and collected in a 250 mL separatory funnel. Juice remaining in the centrifuge bottle was recombined with the juice in the 500 mL separatory funnel and two further separations, each with the addition of 50 g redistilled diethyl ether, were carried out. Solvent collected in each 250 mL separatory funnel was dried over anhydrous sodium sulfate and evaporated to approximately 20 mL on a Calab rotovap (Model 5120, California Laboratory Equipment Co., St. Emeryville, CA) at -55 kPa. The concentrated saskatoon juice solvent extracts were stored in 125 mL test tubes at -23°C.

Saskatoon juice solvent extracts were analysed, as per Leahy and Reineccius (1984) using both a Hewlett Packard 5840A GC/MS 5987 system (Hewlett Packard Corp, Corvallis, OR) fitted with a: 1) slightly polar capillary column (Econo-cap SE54, 30 m X 0.25 mm id X 0.25 μm immobilized 5% diphenyl 95% dimethyl polysiloxane film, Alltech, Deerfield, IL), and 2) a polar capillary column (Supelcowax 10, 30m X 0.25 mm id X 0.25 μm polyethylene glycol bonded film, Supelco Canada, Oakville, ON). Helium, at a velocity of 30 cm/sec, was used as the carrier gas. Separation conditions included an initial oven temperature of 50°C for 2 min and a linear programme of 20°C/min to 275°C for the SE54 column and 15°C/min for the Supelcowax 10 column. A 2μL sample was injected without splitting into an injection port at 220°C. Chromatogram peak areas were recorded as a percentage of that for 2,6-dimethyl phenol. The GC/MS interface and ion source temperatures were 275°C and 200°C, respectively. Mass spectra were obtained with an ionization energy of 70 eV and within the mass range of m/z 40-350. A Hewlett Packard NIH/EPA/MSDC mass spectral database #041 was used for tentative chemical identification of chromatogram peaks. The database provided probabilities for tentative identifications based on the pattern of ion occurrences and relative proportions as compared to the mass spectra pattern in the database. Identifications were confirmed by comparing data with retention times and mass spectra of known standards. GC analyses using both columns were carried out in quadruplicate for each juice treatment within a replicate.

Data for yield, colour, sugars, acids, and areas of 24 peaks from the Supelcowax 10 column and 30 peaks from the SE54 column of all four treatments were subjected to analysis of variance (ANOVA). Sources of variation were : processing treatments (t=4), replications (r=4), instrumental colour (c=6), sugars and acids (s=2), and volatile constituents (v=4). Significant differences ($p \leq 0.05$) among treatments were identified by Student-Newman Keul's multiple range test.

3.3. Results and Discussion

3.3.1. Yield

Mean yields of cold (C), pectinase (P), hemicellulase and pectinase (HP), and hot (H) processed saskatoon juices, from the pulper finisher and after centrifugation are presented in Table 3.1. From the pulper finisher, yield of HP juice was significantly higher ($p \leq 0.05$) than H and P juices, which were similar, and significantly higher ($p \leq 0.05$) than C juice. C and H pulper finisher juice yields were similar to those previously reported (chapter 2); 38.3% for C and 47.0% H juices. The 6% increase in saskatoon juice yield from hot to cold processing is higher than the 2% increases determined for blueberries (Yang, 1984) and cherries (Tressler et al., 1980). Differences in fruits, freezing, and processing conditions may account for the greater increase in yield of hot processed saskatoon juice versus blueberry or cherry juices. Compared to C juice, P and HP pulper finisher juice yields were 4% and 16% greater, respectively. Boyce (1986) reported that enzymes increased yields (6-10%) depending on the type of fruit and enzymes used; HP combinations gave higher yields than P enzymes. Yang (1984) reported a HP treatment

Table 3.1. Means[1] and standard errors for percentage yield of processed saskatoon berry juices.

% Yield	Processed Juice Treatment				SEM[2]
	Cold C	Enzyme P	Enzyme HP	Hot H	
From pulper finisher	40.2^{c}	44.2^{b}	56.1^{a}	46.2^{b}	1.14*
After centrifugation	27.3^{c}	31.3^{b}	25.4^{c}	35.8^{a}	0.88*

[1] Means are the average of four determinations

[2] Standard error of the means

[abc] Means within the same row sharing a common letter are not significantly different at * = $p \leq 0.05$

increased blueberry juice yields by 13% over a C treatment. Results of the present study show similar (4-16%) increases in yields for saskatoon juices using P and HP treatments. Mazza et al. (1978), Mazza (1979), and Mazza (1980), utilizing pectic enzymes and a laboratory hydraulic press, did not determine differences in saskatoon juice yields due to enzymes. The hydraulic press used by Mazza et al. (1978), Mazza (1979), and Mazza (1980) on a small lab sample of saskatoon berries may have created more force on the fruit than the present study's commercial pulper finisher. Thus, the effect of enzymes in disrupting cell structure may not have been as significant a factor to hydraulically pressed juices as to pulper finisher juices.

After centifugation, juice yields for all treatments were reduced. Yield of H juice was significantly higher ($p \leq 0.05$) than P juice, which was also higher than C and HP juices. Centrifugation was used to remove particulate matter and to ensure clarified juices for instrumental and sensory analyses. In C, P, and H clarified juices the particulate matter formed a solid cake on the bottom of the centrifuge bottle and clear juice could be readily separated. HP juices contained fine particulate matter which did not readily form a solid layer. Thus, it was difficult to separate clear HP juice and yields were low. Repeated centrifugation at higher rpm and/or longer times may have improved the yield of HP juice.

3.3.2. Colour

The HunterLab 'L','a', and 'b' values for the four saskatoon juice treatments are presented in Table 3.2. 'L' values of C and P juices were similar and significantly ($p \leq 0.001$) higher than HP juice which was also higher than H juice. There were no significant differences in the 'a' (red) values among the treatments. C and P juices had similar significantly higher ($p \leq 0.01$) 'b' (yellow) readings than H juice. HP juice was similar to all other juices in yellowness.

Table 3.2. Means[1] and standard errors for instrumental colour analyses of processed saskatoon berry juices.

Measurement	Processed Juice Treatment				SEM[2]
	Cold C	Enzyme P	Enzyme HP	Hot H	
'L' value	14.83[a]	14.41[a]	11.56[b]	9.26[c]	0.70***
'a' value	22.50	23.80	22.02	23.18	0.71[ns]
'b' value	8.42[a]	8.37[a]	6.19[ab]	5.27[b]	0.62**

[1] Means are averages of 24 determinations (6 per 4 replicates).

[2] Standard error of the mean

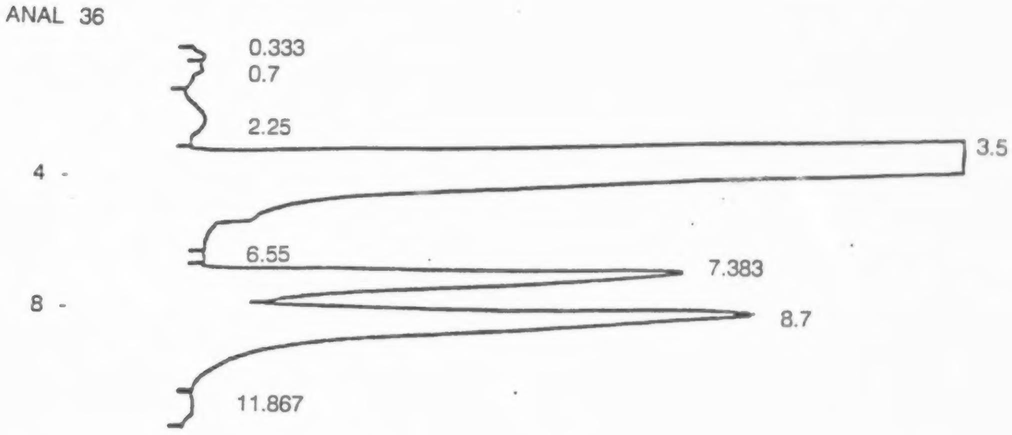
[ns] Not significant

[abc] Means within the same row sharing a common letter are not significantly different at ** = $p \leq 0.01$, and *** = $p \leq 0.001$

Anthocyanins are responsible for the purple/red colour of saskatoon berries (Green and Mazza, 1986). Tannins present in fruit form the substrates for oxidative enzyme reactions which cause lightening and yellowing of colour, creating a brownish appearance, when fruits are juiced (Moyer and Aitken, 1980). Dimick et al. (1951) determined that heating juices to 85°C for 30 sec inactivated the oxidative enzymes which cause browning. The H juice process of 77°C for 5 min was probably sufficient to inactivate the enzymes and prevent browning which occurred in the C, P, and HP saskatoon juices.

3.3.3. Sugars

Glucose and fructose were the only two sugars found in the saskatoon juices. A typical HPLC profile of the sugars present in C saskatoon juice is presented in Figure 3.1. Mean concentrations for glucose and fructose in the juice treatments (Table 3.3) show that H juice had significantly more glucose ($p \leq 0.05$) than C, P, and HP juices which were alike. All juices were similar in fructose content. The combined sugar content of each juice was: C, 14.11%; P, 13.62%; HP, 14.05%; and H, 15.49%. The results of this study are consistent with previous work (chapter two) which showed the soluble solids content of C juice at 16.0% and H juice at 16.7%.



Retention time 7.383 min = fructose
Retention time 8.7 min = glucose

Figure 3.1. HPLC profile of sugars in cold (3°C) processed saskatoon juice.

Table 3.3. Means[1] and standard errors for percentages of glucose and fructose in processed saskatoon berry juices.

Sugars[3]	Processed Juice Treatment				SEM[2]
	Cold C	Enzyme P	Enzyme HP	Hot H	
Glucose	8.25^{b}	7.91^{b}	8.24^{b}	9.26^{a}	0.25^{*}
Fructose	5.86	5.71	5.81	6.23	0.18^{ns}

[1] Means are the averages of 8 determinations (2 per 4 replicates).

[2] Standard error of the mean

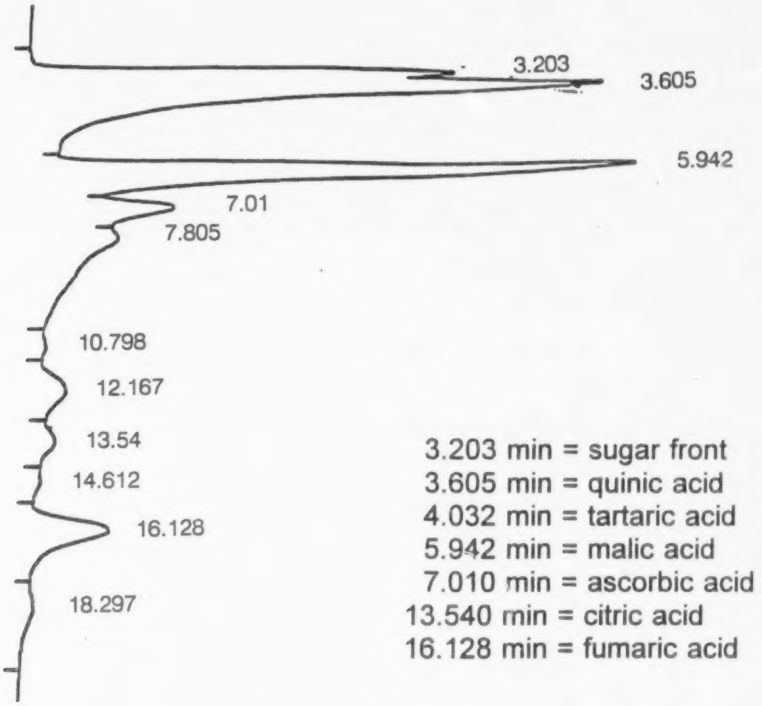
[3] Grams sugar per 100 grams juice

[ns] Not significant

[abc] Means within the same row sharing a common letter are not significantly different at * = $p<0.05$

3.3.4. Organic Acids

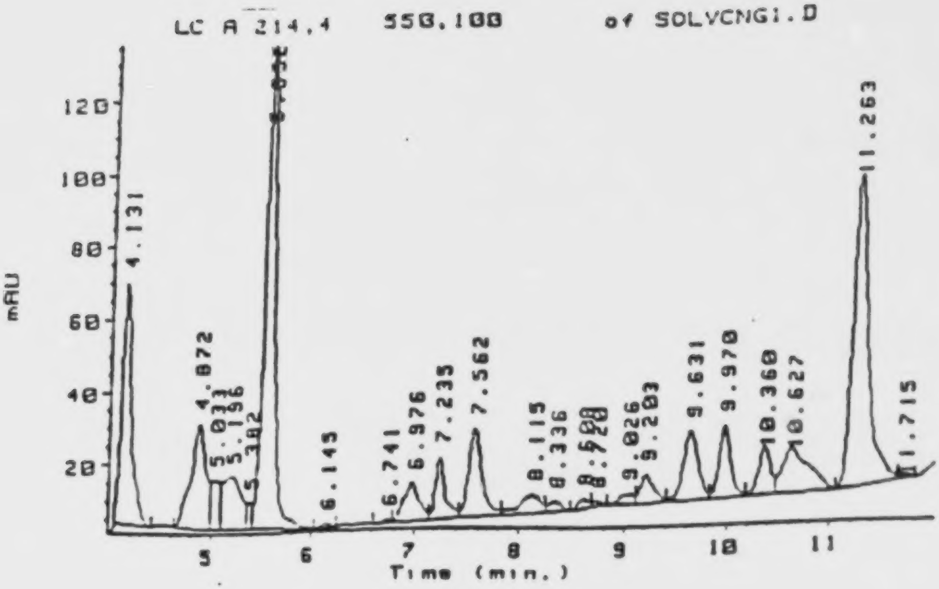
A typical HPLC profile of organic acids based on the ion-exchange method of Coppola and Starr (1986) is presented in Figure 3.2. The sugar front (unretained on column) appeared at 3.2 min with quinic acid at 3.6 min; both were not resolved sufficiently to allow quantification. The quinic acid peak tail also extended into the elution of the tartaric acid standard (4.0 min). Coppola and Starr (1986) acknowledged difficulties in separating early eluting organic acids, especially quinic. Further efforts using this technique were not pursued.



3.203 min = sugar front
3.605 min = quinic acid
4.032 min = tartaric acid
5.942 min = malic acid
7.010 min = ascorbic acid
13.540 min = citric acid
16.128 min = fumaric acid

Figure 3.2. HPLC profile of organic acids in hot processed saskatoon juice.

A typical HPLC profile of organic acids based on the ion-pairing method of Simpson (1982) is shown in Figure 3.3. This method successfully separated the sugar front from quinic acid and clearly resolved ascorbic, citric, and fumaric acids, previously not found in saskatoon juice. However, malic acid, previously identified (Wolfe and Wood, 1971) in mature saskatoon berries was not well resolved. The tartaric acid standard had the same retention time as quinic acid. Coppola and Starr (1986) reported quinic but not tartaric acid in



4.872 min = quinic + tartaric
5.382 min = malic
5.532 min = ascorbic
7.235 min = citric
7.562 min = fumaric

Figure 3.3. HPLC profile of organic acids in cold (3°C) processed saskatoon juice.

cranberries. Based on results from the ion exchange method (Figure 3.2), it appears that if tartaric acid is present within the quinic acid peak it is probably only a small percentage of the overall peak area. Means for organic acid content of the four saskatoon juice treatments are presented in Table 3.4. P juice contained significantly ($p<0.05$) more fumaric acid than C, HP, and H juices which were similar. The quantity of fumaric acid in saskatoon juices is very low; approximately two orders of magnitude less than other organic acids present.

Table 3.4. Means[1] and standard errors for organic acids in processed saskatoon berry juices.

Organic Acids[3]	Processed Juice Treatment				SEM[2]
	Cold C	Enzyme P	Enzyme HP	Hot H	
Ascorbic	2.89	2.66	2.80	3.73	0.29[ns]
Citric	5.68	5.87	7.18	7.32	0.57[ns]
Fumaric	0.07[b]	0.18[a]	0.09[b]	0.09[b]	0.02*
Malic	3.12	6.20	4.98	3.96	1.17[ns]
Quinic + Tartaric[5]	36.83	36.75	52.21	45.15	5.04[ns]

[1] Means are the averages of 8 determinations (2 per 4 replications)
[2] Standard error of the mean
[3] Milligrams acid per 10 grams juice
[4] Quantified according to 0.2% quinic acid standard
[ns] Not significant
[abc] Means within the same row sharing a common letter are not significantly different at * = $p \leq 0.05$

There were no significant differences in ascorbic, citric, malic, and quinic/tartaric acids in the C, P, HP, and H juices. Quinic acid has not been previously reported in saskatoon berries but values 2 - 3 times higher have been reported in cranberries (Coppola and Starr, 1986). Unfortunately, reproducibility of the results was a problem, possibly due to the column age and difficulties in maintaining constant pressure. Thus, the variation within treatments was large, contributing to a large statistical error term. Data for malic are of concern due to the lack of consistency with other studies. Wolfe and Wood (1971) determined malic acid content of saskatoon berries to be 2.5 - 8.5 times higher than those determined in the present study. The results of Wolfe and Wood (1971) were similar to those found by Coppola and Starr (1986) for other rosaceous fruits. As the malic acid peak in the present study was not resolved well by ion-pairing, the methods of Wolfe and Wood (1971) or Coppola and Starr (1986) using ion-exchange may be better for quantifying malic acid content in saskatoon juice.

3.3.5. Volatile Constituents

Typical GC profiles of volatile constituents present in C juice, as determined by SE54 and Supelcowax 10 columns, are presented in Figures 3.4 and 3.5, respectively. The GC chromatogram from the SE54 column had 5 peaks with confirmed identities, 9 peaks with tentative (MS library database) identities, and 16 unidentified peaks. The chromatogram from the Supelcowax 10 column had 3 confirmed, 5 tentative, and 15 unidentified peaks. Although it gave fewer reproducible peaks than the SE54 column, the Supelcowax 10 column was capable of resolving more early eluting, low molecular weight, volatiles which often have a significant impact on flavour. GC profiles of P, HP, and H juices showed similar peaks but their proportions varied.

1. unknown
2. benzaldehyde (C)
3. phenol (T)
4. benzyl alcohol (C)
5. phenyl acetaldehyde (T)
6. linalool oxide (T)
7. methyl benzoate (T)
8. 2,6 dimethyl phenol (standard)
9. 2-phenyl ethanol (C)
10. benzoic acid (C)
11. mandelonitrile (T)
12. eugenol (T)
13. butyl butanoate (T)
14. dodecanol (T)

15. - 16. unknown

17. diphenol methanone (T)

18. - 20. unknown

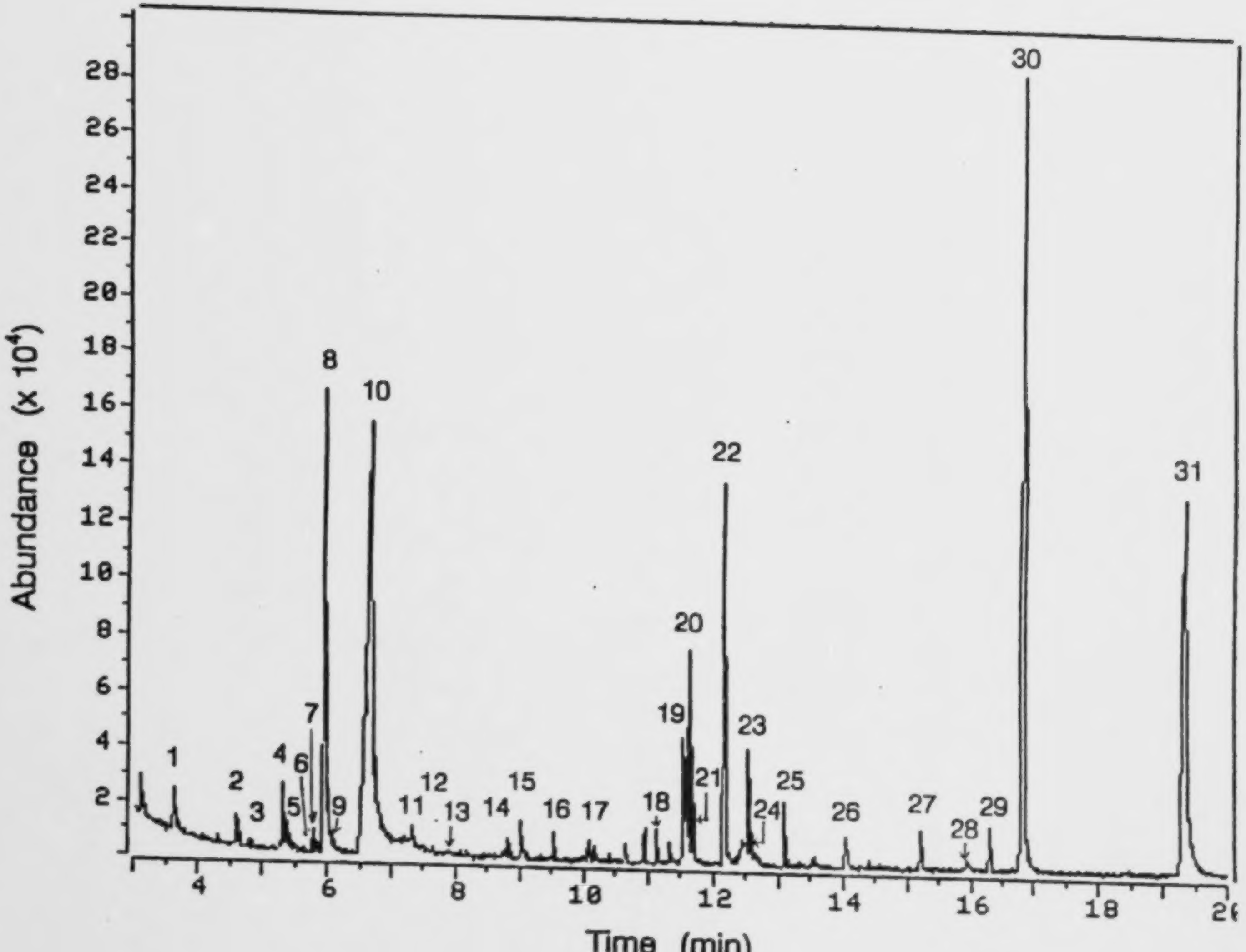
21. ethyl hexadecanoate (C)

22. - 31. unknown

(T) = tenative identification

(C) = confirmed identification

Figure 3.4. GC (SE54 column) profile of volatile constituents in cold (3°C) processed saskatoon juice.


Abundance (x 10⁴)
28
26
24
22
20
18
16
14
12
10
8
6
4
2
Time (min)
4
6
8
10
12
14
16
18
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20
21
22
23
24
25
26
27
28
29
30
31

1. - 5. unknown

6. Z-pentene-1-ol (T)

7. linalool oxide (T)

8. benzaldehyde (C)

9. unknown

10. phenyl acetaldehyde (T)

11. - 13. unknown

14. benzyl alcohol (C)

15. 2,6 dimethyl phenol (standard)

16. - 18. unknown

19. ethyl hexadecanoate (C)

20. - 21 unknown

22. octadecanol (T)

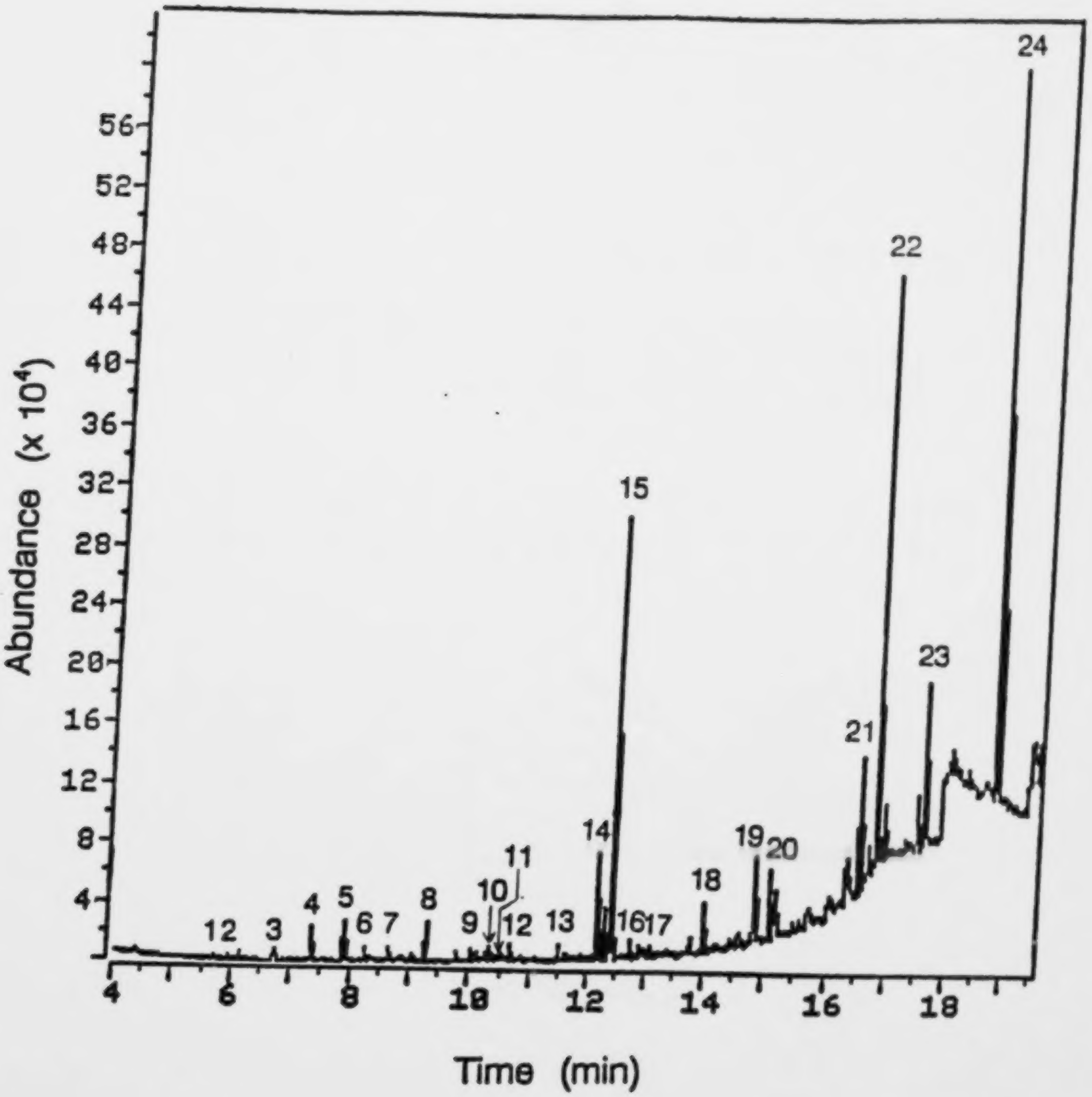
23. 1,2-benzene dicarboxylic acid (T)

24. octacosane (T)

(T) = tenative identification

(C) = confirmed identification

Figure 3.5. GC (Supelcowax 10 column) profile of volatile constituents in cold (3°C) processed saskatoon juice.


Abundance (x 10⁴)
56
52
48
44
40
36
32
28
24
20
16
12
8
4
Time (min)
4
6
8
10
12
14
16
18
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20
21
22
23
24

Octacosane was the only aliphatic hydrocarbon tentatively (p=0.89) identified in saskatoon juice using the Supelcowax 10 column. Octacosane has been identified in plum extracts (Ismail, 1977; Vernin et al., 1985).

Aliphatic alcohols tentatively identified in the saskatoon juices included dodecanol (p=0.93 / SE54 column), Z-pentene-1-ol (p=0.70 / Supelcowax 10 column), Z-hex-3-en-1-ol (p=0.76 / Supelcowax 10 column) and octadecanol (p=0.92 / Supelcowax 10 column). Dodecanol has been reported in strawberries (Drawert, 1970), Z-pentene-1-ol in peaches (Spencer et al., 1978) and raspberries (Winter and Enggist, 1971), Z-hex-3-en-1-ol in mangoes (Engel and Tressl, 1983) and octadecanol in custard apple (Wyllie et al., 1987).

Aromatic compounds found in saskatoon juices by one or both GC analyses included benzaldehyde, phenylacetaldehyde (p=0.81), mandelonitrile (p=0.86), benzyl alcohol, and 2-phenyl ethanol. Amygdalin in fruit seeds or stones results in mandelonitrile via glucosidase hydrolysis and benzaldehyde through the subsequent action of hydroxynitrile lyase (Broderick, 1975). Benzyl alcohol and phenyl ethanol were also released through enzymatic hydrolysis of glycosidically bound volatiles (Salles et al., 1988). Benzaldehyde, benzyl alcohol, phenyl ethanol, and phenyl acetaldehyde were identified in pome, stone fruits, or berries such as apples (Dirinck et al., 1977), quinces (Schreyen et al., 1979), apricots (Rodriguez et al., 1980; Guichard and Souty, 1988), plums (Dirninger-Rigo, 1987; Ismail, 1977), peaches (Soboleva and Yazvinskaya,1975; Maarse, 1984), cherries (Broderick, 1975), strawberries (Pyysalo et al., 1979; Schreier, 1980), blueberries (Hirvi and Honkanen, 1983), and cranberries (Anjou and von Sydow, 1967). Mandelonitrile, a very unstable compound, does not appear in the literature as a volatile flavour constituent of the above noted fruits. (Attempts to obtain retention times for mandelonitrile standards resulted in the production of benzaldehyde.) Benzaldehyde, benzyl alcohol, phenyl ethanol, benzoic acid, and methyl benzoate may be products of the Shikimic acid pathway (Pyysalo, 1976). Benzoic acid and methyl benzoate ($p=0.97$) were determined in saskatoon juice by the SE54 column analysis. Both benzoic acid and methyl benzoate were found in cranberries (Croteau and Fagerson, 1968).

Two phenolic compounds, phenol and eugenol (2-methoxy-4-(2-propenyl)phenol), were tentatively identified ($p=0.86$ and 0.95, respectively) in the SE54 column analysis. Phenol has been found in cherries (Broderick, 1975), blueberries (Hirvi and Honkanen, 1983), and cranberries (Anjou and von Sydow, 1967). Eugenol is characteristic of cloves and has been determined in quinces (Umano et al., 1986), strawberries (Pyysalo et al., 1979), blueberries (Hirvi and Honkanen, 1983), and cranberries (Hirvi et al., 1981).

Terpene alcohols tentatively identified in saskatoon juices include linalool oxide ($p=0.76$) and α-terpineol ($p=0.91$) by both the SE54 and Supelcowax columns. α-Terpineol had a retention time similar to the 2,6-dimethyl phenol standard and thus could not be quantified. Analysis without the additon of the standard indicated that the α-terpineol peak was < 1% of the benzaldehyde peak area. Linalool oxides and α-terpineol have been found in apples (Yajima et al., 1984), quinces (Tsuneya et al., 1983), plums (Ismail, 1977), peaches (Souty and Reich, 1978), and strawberries (Pyysalo et al., 1979; Schreier, 1980).

Two esters were identified in saskatoon juices: ethyl hexadecanoate (both SE and Supelcowax 10 column), and butyl butanoate (p=0.81 / SE54 column). Ethyl hexadecanoate has been determined in pears (Jennings and Tressl, 1974), quinces (Umano et al., 1986), apricots (Rodriguez et al., 1980), and blackberries (Georgilopoulos and Gallois, 1987). Esters may be derived from fatty acid precursors during ripening. In apple pulp, hexadecanoic acid, which comprises 31% of the total fatty acids, may be the precursor of ethyl hexadecanoate (Galliard, 1968). Butyl butanoate has been found in apples (Drawert et al., 1969), quinces (Umano et al., 1986), plums (Dirninger-Rigo, 1987; Ismail, 1977), strawberries (Drawert, 1970; Schreier, 1980), and blackberries (Georgilopoulos and Gallois, 1987).

A ketone, diphenol methanone (p=0.95 / SE54 column) and an organic acid, phthalic acid (1,2 benzene dicarboxylic acid) (p=0.95 / Supelcowax 10 column), were also tentatively identified in saskatoon juices. To date, these compounds have not been reported in the literature in other fruits or berries.

Mean identified peak areas, for identified volatiles, for each of the four processed saskatoon juice treatments, are presented in Tables 3.5 (SE54 column) and 3.6 (Supelcowax 10 column).

The aliphatic hydrocarbon octacosane (Table 3.6) was present in significantly ($p \leq 0.001$) higher concentration in C than H and P juices which were similar and also higher than in HP juice. Cellulitic enzymes appear to alter the structure of octacosane but compounds resulting from its breakdown have not been determined. Octacosane has not been identified in the literature as a significant aroma compound in fruits.

C juice contained the highest levels of the three aliphatic alcohols found in all treatments . C juice had significantly ($p \leq 0.01$) more dodecanol (Table 3.5) than P and H juices which were similar and had significantly more than HP juice. C juice contained significantly ($p \leq 0.001$) higher amounts of Z-pentene-1-ol (Table 3.6) as compared to H juice which contained higher amounts than P and HP juices which were alike. C and H juices contained similar ($p \leq 0.001$) greater quantities of octadecanol (Table 3.6) than P and HP juices which were similar. Of these three aliphatic alcohols only dodecanol has been found to contribute to aroma. Idstein and Schreier (1985) determined that dodecanol had a herbaceous, violet-like aroma. The reduced levels of dodecanol in H, P, and HP juices compared to C juice may affect the aroma of these juices. One other aliphatic alcohol, Z-hex-3-en-1-ol (Table 3.6), was found in measurable

Table 3.5. Means[1] and standard errors for GC analysis (SE54 column) of identified volatiles in saskatoon berry juices.

Volatiles	Processed Juice Treatment				SEM[2]
	Cold C	Enzyme P	Enzyme HP	Hot H	
benzaldehyde	6.46^{c}	7.68^{c}	26.94^{b}	427.69^{a}	30.54***[3]
phenol	2.39	2.39	0.83	2.34	0.59^{ns}
benzyl alcohol	21.78^{d}	137.81^{b}	194.19^{a}	94.39^{c}	7.17***
phenyl acetaldehyde	6.96	4.87	4.27	5.04	1.35^{ns}
linalool oxide	3.39	4.01	3.83	3.53	0.28^{ns}
methyl benzoate	1.45^{b}	1.99^{a}	2.23^{a}	1.06^{b}	0.12**
2-phenyl ethanol	5.21^{b}	9.35^{a}	11.73^{a}	11.96^{a}	1.28***
benzoic acid	375.63^{b}	498.94^{a}	518.69^{a}	228.19^{c}	19.57***
mandelonitrile	0.73^{b}	8.07^{a}	1.80^{b}	6.31^{a}	0.73**
eugenol	0.88^{b}	1.27^{b}	4.57^{b}	12.21^{a}	1.63**
butyl butanoate	0.84^{b}	0.60^{b}	0.69^{b}	3.44^{a}	0.18**
dodecanol	4.50^{a}	1.38^{b}	0.59^{c}	1.96^{b}	0.60**[3]
diphenol methanone	3.05^{a}	1.09^{c}	0.81^{c}	1.99^{b}	0.28***
ethyl hexadecanoate	11.06^{a}	3.01^{b}	1.82^{b}	2.81^{b}	1.09***

[1] Means are the average of 16 determinations (4 per 4 replicates) expressed as percentage of internal standard

[2] Standard error of the mean

[3] Analysis based on $\log_{10}$ of data (Montgomery, 1984)

[abc] Means within the same row sharing a common letter are not significantly different at ** = $p \leq 0.01$ and *** = $p \leq 0.001$

Table 3.6. Means[1] and standard errors for GC analysis (Supelcowax 10 column) of identified volatiles in saskatoon berry juices.

Volatiles	Processed Juice Treatment				SEM[2]
	Cold C	Enzyme P	Enzyme HP	Hot H	
Z-pentene-1-ol	4.09^{a}	0.80^{c}	0.50^{c}	1.57^{b}	0.60^{***}[3]
linalool oxide	4.17	4.03	4.15	3.78	0.13^{ns}
benzaldehyde	8.12^{d}	10.66^{c}	31.75^{b}	393.44^{a}	28.62^{***}[3]
Z-hex-3-en-1-ol[6]	<0.50	1.94	<0.50	<0.50	
phenyl acetaldehyde	3.76^{a}	2.05^{b}	2.34^{b}	2.89^{b}	0.24^{**}
benzyl alcohol	27.80^{d}	148.38^{b}	205.94^{a}	102.82^{c}	6.95^{***}
ethyl hexadecanoate	13.27^{a}	4.02^{b}	2.64^{b}	3.82^{b}	0.93^{***}
octadecanol	77.43^{a}	21.79^{b}	12.13^{b}	60.25^{a}	8.71^{***}
phthalic acid[5]	55.80^{a}	15.00^{b}	11.50^{b}	22.50^{b}	5.17^{***}
octacosane	280.26^{a}	144.93^{b}	69.74^{c}	180.48^{b}	21.27^{***}

---

[1] Means are the average of 16 determinations (4 per 4 replicates) expressed as percentage of internal standard unless indicated as in [5] and [6]

[2] Standard error of the mean

[3] Analysis based on $\log_{10}$ of data (Montgomery, 1984)

[4] Compound only found above detection limit (0.5%) in treatment P

[5] Compound only found above detection limit for 7 determinations of P and 3 determinations of HP

[ns] Not significant

[abc] Means within the same row sharing a common letter are not significantly different at ** = $p \leq 0.01$ and *** = $p \leq 0.001$

quantities in P juice only. This compound has been found to have a green, grassy, fruity aroma (Engel and Tressl, 1983).

Levels of aromatic compounds differed significantly among juice treatments (Tables 3.5 and 3.6). Benzaldehyde was low in C and P juices. HP juices contained approximately 3X more benzaldehyde than C and P juices. H juices contained 40-50X more benzaldehyde than C and P juices (Supelcowax 10 and SE54 column analysis, respectively). Although analyses via both columns gave the same order of magnitude of differences in benzaldehyde among the treatments, with the Supelcowax 10 column P juice contained significantly ($p \leq 0.001$) more benzaldehyde than C juice whereas with the SE54 column a significant difference was not found. This lack of significance was likely due to a greater variation within treatments for SE54 column data than for Supelcowax 10 data. Production of benzaldehyde from amgydalin is promoted by warm temperatures and increasing concentrations of enzymes and substrates (Whitaker, 1972a and 1972b). Saskatoon juice treatments were processed at different temperatures : C, 3°C; P, 15°C; HP, 22°C; and H, 77°C. Benzaldehyde levels in the juices increased according to treatment temperature. The P treatment removed the seeds, the major source of amygdalin, prior to enzyme addition. For the HP method, whole berries were ground and the enzyme was added for one hour prior to seed removal. Thus, the HP method probably promoted enzyme/substrate contact and resulted in increased benzaldehyde. The commercial enzymes used may also have **β**-

glucosidase as an impurity which would promote benzaldehyde production. During H processing, the fruit was ruptured releasing enzymes and juice. Both H and HP juices permitted enzyme/substrate contact but H juice had greater than 10X more benzaldehyde than HP juice. The effect of temperature was therefore more significant than the effect of enzyme/substrate contact on benzaldehyde content. Benzaldehye imparts a sweet almondy aroma (Dravnieks, 1985). Benzaldehyde increases, of up to 50X in H juice than in C, P, and HP juices, may thus increase sweet almondy notes in the H saskatoon juice. Mandelonitrile content was significantly ($p \leq 0.01$) higher in P and H juices as compared to C and HP juices. This result does not correspond to benzaldehyde concentration among the four juices. Previous research (chapter two) found no significant differences between mandelonitrile contents of C and H juices. The instability of mandelonitrile (the precursor of benzaldehyde) may account for the differences between results reported in Chapters 2 and 3 as well as mandelonitrile's small peak area in relation to the benzaldehyde peak area.

Data for benzyl alcohol (Tables 3.5 and 3.6) were similar. Results showed significantly ($p \leq 0.001$) more benzyl alcohol in HP than P juices, P than H juices, and H than C juices. Schreier et al. (1978) also found that cellulitic enzymes encouraged the formation of C_6-alcohols in apple juices. Chariote et al. (1981) and Schmid and Grosche (1986) found hot processed apricots and cherries had increased concentrations of aromatic alcohols. Results of the present study agree with findings for other fruits which indicate that enzyme processing may encourage alcohol formation to a greater extent than heat processing. Benzyl alcohol may not be important to fruit aroma due to its low specific odour intensity, but may contribute tartness (Croteau and Fagerson, 1968). Thus, differences in benzyl alcohol content of saskatoon juices may not affect the flavour greatly.

Another aromatic alcohol, 2-phenyl ethanol, was significantly ($p \leq 0.001$) lower in C than in P, HP, and H juices. The increases in 2-phenyl ethanol were not as great as for benzyl alcohol. In apricot pulp, phenyl ethanol was released via enzymatic hydrolysis of glycosidically bound volatiles (Salles et al., 1988). 2-Phenyl ethanol has been described as fragrant, floral and sweet (Dravnieks, 1985) and rose/honey-like (Stevens et al., 1970). Thus, low levels of 2-phenyl ethanol may influence the flavour of cold saskatoon juice.

Phenyl acetaldehyde levels did not differ significantly among the four saskatoon juice treatments acccording to SE54 column analysis. However, via the Supelcowax column analysis, phenyl acetaldehyde was significantly ($p \leq 0.01$) higher in C than in P, HP, and H juices. Both columns gave similar trends in the means of the four treatments but the SE54 column data varied within a treatment too much to provide statistically significant results. 2-Phenyl acetaldehyde is characterised by a green, floral, sweet aroma (Engel and Tressl, 1983). A reduction in this compound may contribute to a loss of these flavour characteristics in P, HP and H saskatoon juices.

Differences in benzoic acid levels were observed among the four juice treatments. P and HP juices had significantly ($p \leq 0.001$) more benzoic acid than C juice which also had more than H juice. Benzoic acid accounted for approximately 27% of the total peak area on the C juice chromatograms. Croteau and Fagerson (1968) also reported similar findings. However, as there were no differences in aromas of total and acid-free extracts, the authors concluded that benzoic acid was not a significant odour component. Methyl benzoate levels in saskatoon juices followed the same trend as those of benzoic acid with HP > P > C > H juice. P and HP juices had significantly ($p \leq 0.01$) higher methyl benzoate than C and H juices. These results may be expected as methyl benzoate may be derived from benzoic acid (Pyysalo, 1976) Methyl benzoate has a pungent, heavy, sweet, floral character (Idstein and Schreier, 1985) which may influence saskatoon juice flavour.

Of the two phenolic compounds, phenol did not differ due to treatment while eugenol was significantly ($p \leq 0.01$) higher in H juices than in C, P, and HP juices. Phenol is characterised as medicinal (Idstein and Schreier, 1985); eugenol is described as sweet and spicy (cloves) (Dravnieks, 1985). The greater than ten fold increase in eugenol in H juice versus C juice may impact on the aroma profile and strength of the H juice.

Linalool oxide, the only quantifiable terpene alcohol, was determined by both columns but not found to differ significantly among the juice treatments. Linalool oxide aroma is sweet, woody, and earthy (MacLeod and Pieris, 1983).

Analyses of the ester ethyl hexadecanoate by both columns gave significantly ($p \leq 0.001$) higher amounts in C than P, HP, and H juices which were similar. This ester contributes a faint waxy creamy aroma to fruits (Idstein and Schreier, 1985) but is not considered to be a strong aroma impact compound. Thus, different concentrations of ethyl hexadecanoate may not significantly affect the resulting aroma of the four saskatoon juice treatments. The other ester, butyl butanoate, was significantly ($p \leq 0.01$) higher in H than in C, P, and HP juices which did not differ. This ester has fresh, sweet, and fruity notes (Flath and Forrey, 1977) and is important to pear aroma (Chua et al., 1987). Thus, the four fold increase in butyl butanoate in H juice versus C, P, and HP juices may significantly affect H juice aroma.

Diphenol methanone concentration was significantly ($p \leq 0.001$) higher in C juice than H juice, which was also higher than in P and HP juices which were similar. The level of phthalic acid was significantly ($p \leq 0.001$) higher in C juice than P, HP, and H juices which are alike. Neither of these two compounds has been reported as contributing to fruit aroma; thus, their high levels in C juice may not affect its aroma.

Mean peak areas for unidentified volatiles for each of the four processed saskatoon juice treatments are presented in Table 3.7 (SE54 column) and Table 3.8 (Supelcowax 10 column). GC data are the average of sixteen determinations; however for some juice treatments complete data were not available. For example, peak number 21 (Table 3.8) was consistently present

Table 3.7. Means[1] and standard errors for GC analysis (SE54 column) of unidentified volatiles in saskatoon berry juices.

Peak No.	Processed Juice Treatment				SEM[2]
	Cold C	Enzyme P	Enzyme HP	Hot H	
1	10.75^{a}	10.77^{a}	11.04^{a}	4.97^{b}	0.43***
15	13.11^{a}	1.79^{c}	1.47^{c}	5.50^{b}	2.05^{**3}
16	6.58^{a}	6.16^{a}	4.29^{b}	5.38^{ab}	0.48*
18	3.95^{a}	0.76^{c}	0.62^{c}	1.24^{b}	0.50^{**3}
19	21.66^{a}	6.31^{b}	5.21^{b}	4.83^{b}	2.28***
20	42.76^{a}	6.01^{c}	3.71^{c}	21.18^{b}	6.64^{**3}
22	36.46^{a}	10.44^{b}	6.42^{b}	30.89^{a}	3.98***
23	13.22^{a}	3.94^{b}	1.98^{c}	1.73^{c}	1.24^{***3}
24	6.99^{a}	1.35^{b}	1.45^{b}	0.70^{b}	0.64***
25	6.75^{a}	2.06^{c}	1.19^{c}	4.41^{b}	0.51***
26	6.35^{a}	2.09^{bc}	1.32^{c}	3.24^{b}	0.42***
27	8.14^{a}	2.46^{b}	1.59^{b}	3.82^{b}	0.70***
28	6.94	4.10	5.59	2.44	1.58^{ns}
29	13.03^{a}	2.34^{b}	2.09^{b}	1.81^{b}	0.98***
30	240.05^{a}	92.49^{bc}	41.42^{c}	98.08^{b}	20.28^{***3}
31	212.67^{a}	64.50^{b}	24.15^{b}	100.51^{b}	25.18**

[1] Means are the average of 16 determinations (4 per 4 replicates) expressed as percentage of internal standard

[2] Standard error of the mean

[3] Analysis based on $\log_{10}$ of data (Montgomery, 1984)

[ns] Not significant

[abc] Means within the same row sharing a common letter are not significantly different at : $* = p \leq 0.05$, $** = p \leq 0.01$, and $*** = p \leq 0.001$

Table 3.8. Means[1] and standard errors for GC analysis (Supelcowax 10 column) of unidentified volatiles in saskatoon berry juices.

Peak No.	Processed Juice Treatment				SEM[2]
	Cold C	Enzyme P	Enzyme HP	Hot H	
1	2.04^{b}	3.52^{a}	2.05^{b}	1.61^{c}	0.12^{***}
2	1.42^{12}	0.71^{11}	0.78^{14}	0.97	0.24^{ns}
3	4.60^{b}	12.22^{a}	3.93^{b}	4.84^{b}	0.78^{***}
4	6.75^{a}	7.14^{a}	7.41^{a}	3.41^{b}	0.23^{***}
5	10.58^{a}	7.85^{b}	8.25^{b}	5.31^{c}	0.35^{***}
9	2.61	2.28	3.39	2.59	0.29^{ns}
11	2.06^{a}	1.97^{a}	2.47^{a}	1.19^{b}	0.15^{***}
12	2.64^{a}	0.92^{b}	0.68^{b}	1.48^{b}	0.24^{***}
13	2.76^{a}	1.34^{c}	0.95^{c}	1.94^{b}	0.18^{***}
16	6.05	4.75	4.62	4.95	0.45^{ns}
17	4.35^{a}	3.34^{ab}	1.99^{b}	3.55^{ab}	0.50^{*}
18	2.21^{a15}	1.20^{b10}	0.95^{b6}	1.50^{b15}	0.21^{**}
20	12.51^{a}	8.88^{ab}	7.01^{b}	11.18^{a}	1.11^{*}
21	24.39^{a}	8.49^{b14}	4.71^{c11}	3.62^{c12}	1.17^{***}

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[1] Means are the average of 16 determinations (4 per 4 replicates) expressed as percentage of internal standard unless otherwise indicated by number after letter (eg. a12 = 12 determinations were > detection limit of 0.5%)

[2] Standard error of the mean

[ns] Not significant

[abc] Means within the same row sharing a common letter are not significantly different at: * = $p \leq 0.05$, ** = $p \leq 0.01$, and *** = $p \leq 0.001$

at 24.39% of the internal standard in C juice but was reduced to 3.62% in H juice and was only detected in 12 of 16 determinations. Although incomplete, these data have been included to indicate changes in aroma compounds among juice treatments.

Sixteen unidentified GC peaks were present in SE54 column analysis of saskatoon juices. Peak 1 in C, P, and HP juices (Table 3.7), the only early eluting low molecular weight compound, was significantly ($p \leq 0.001$) higher than in H juice. The heat treatment may have volatilized some of this low molecular weight compound. The other unidentified peaks (SE54 column) 15, 16, 18-20, and 22-31 are in general high molecular weight compounds. Compared to C juice the total unidentified peak areas of P, HP, and H juices were reduced by 66%, 82%, and 55% , respectively. These significant reductions in volatiles may affect aroma of the saskatoon juices.

Fourteen GC peaks were unidentified via the Supelcowax 10 column analysis of saskatoon juices (Table 3.8). Peaks 1 - 5, low molecular weight compounds, may be readily vaporised and contribute significantly to aroma of saskatoon juices. P juice contained significantly ($p \leq 0.001$) higher amounts of peaks 1 and 3 than C, HP, and H juices. These compounds may be breakdown products of higher molecular weight components hydrolysed by the enzymes. Compared to C juice the total unidentified volatile peak area of P, HP, and H juices was reduced 32%, 48%, and 49%, respectively. This reduction in volatile constituents may affect aroma intensity of the juices.

Results of this study show that the processing method used to extract saskatoon berry juice (cold, pectinase, pectinase + hemicellulase, or hot) had significant effects on juice yield, colour, sugar, acid, and volatile constituents. Two alternatives for high yields were determined depending on the characteristics of the juice desired for further processing. The HP process produced a high yield of juice with fine particulate matter and a lighter and more yellow colour than H processed juice. The H process gave a high yield of centrifuged clear juice. H juice had significantly higher glucose than C, P, and HP juices while all juices had similar fructose contents. Acids determined in saskatoon juices included malic, citric, quinic, fumaric, and ascorbic acids. The latter three, including the predominant quinic acid, have not previously been reported in saskatoon berries. Generally the acid content among the treatments did not vary. Eighteen volatile constituents were identified in saskatoon juices and thirty other volatiles were recorded via the GC analyses. The quantities of identified and unidentified volatile constituents in the four saskatoon juice treatments suggest that the aroma profiles are different. Reductions in many aroma compounds in P, HP, and H juices as compared to C juice may alter many significant "fresh" fruit components, but increases in aromatics such as benzaldehyde and benzyl alcohol may provide new aroma characteristics for P, HP, and H juices. Further research is necessary to determine the effects of these chemical constituents on the sensory attributes of the four saskatoon juice treatments.

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Chapter 4 Influence of Saskatoon Berry Seeds on Three Aromatic Compounds in Processed Saskatoon Berry Juice

4.1. Introduction

Benzaldehyde was determined to be a major aroma component of saskatoon berries by Mazza and Hodgins (1985). Broderick (1975) demonstrated that benzaldehyde in cherry juice was a breakdown product of amygdalin present in cherry seeds. Majak et al. (1981) found amygdalin in the fruit and foliage of saskatoons. Thus, benzaldehyde found in saskatoon juice (Mazza and Hodgins, 1985) may have resulted from the breakdown of amygdalin in the seeds.

Cold extracted cherry juices had less benzaldehyde and a weaker flavour than hot extracted cherry juices (Schmid and Grosche, 1986) . Recent work (chapter three) on saskatoon juice showed low concentrations of benzaldehyde in solvent extracts of cold (3°C) processed juices and higher levels (40-50X) in solvent extracts of hot (77°C) processed juices. Benzoic acid was present in both the hot and cold juices. Hot saskatoon juice had higher levels of benzyl alcohol than cold juice. Hatanaka and Kaneda (1985) reported that benzaldehyde, produced during the processing of apricot juice, was oxidized to benzoic acid within four hours. Albagnac and Vangheesdaele (1975) determined that benzaldehyde in crushed fermenting cherries was completely reduced to benzyl alcohol in three days. Each of these three aromatic compounds may have a significant effect on saskatoon juice flavour.

To process saskatoon juice of consistent quality, information on the efficacy of temperature and time on the production of benzaldehyde, benzoic acid, and benzyl alcohol is needed.

This study was conducted to determine the individual effects of seeds, temperature, and time on the level of benzaldehyde, benzoic acid, and benzyl alcohol in saskatoon juices.

4.2. Materials and Methods

Three experiments were conducted : 1) juice without seeds (no seeds/NS) was heated from 15°C to 85°C; 2) berries and water were crushed to form a juice with seeds [seeds + water (SW)] and heated from 15°C to 85°C; and 3) whole berries with water were distilled from 0.5 hr to 3 hr. Levels of benzaldehyde, benzoic acid, and benzyl alcohol in juices were determined in all experiments. These experiments were conducted only once due to the limited amount of fruit remaining after Chapter 3 experiments were completed.

Experiment 1: A total of 14 kg of thawed saskatoon berries (3°C) were juiced in a Champion juicer (Model G5NG853S) to remove seeds (NS). The juice was centrifuged (Damon CRU-5000, International Equipment Co., Needham, MA) in 1 L bottles at 1,500 rpm for 10 min and the clarified juice was decanted. Eight lots of 700 g saskatoon juice were weighed into 2 L Erlenmeyer flasks and randomly assigned to heating (on hot plates) to one of the following temperatures : 15, 25, 35, 45, 55, 65, 75, or 85±1°C. When the desired temperature was reached the samples were cooled in an ice water bath to 5°C.

Experiment 2: In a 4 L beaker a total of 800 g water (5°C) and 800 g frozen saskatoon berries were crushed using a polytron blender (Brinkmann PT10/35, Brinkmann Instrument Co., Rexdale, ON) at speed six for 30 sec. Eight samples of SW juice were prepared using this procedure and randomly assigned to heating on hot plates to one of the following temperatures : 15, 25, 35, 45, 55, 65, 75, or 85±1°C. When the desired temperature was reached the samples were cooled in an ice water bath to 5°C. Juice was centrifuged (Damon CRU-5000) in 1 L bottles at 1,500 rpm for 10 min and the clarified juice was decanted.

Experiment 3: The distillation procedure of Mazza and Hodgins (1985) was used as a basis for determining the effect of time on benzaldehyde, benzyl alcohol, and benzoic acid production in saskatoon juice. A total of 250 g frozen saskatoon berries and 250 g distilled water were combined in a 1 L boiling flask with boiling chips and connected to distillation apparatus as shown in Appendix 1. The vertical condensor contained 2 mL hexane for collection of volatile constituents. The hexane was removed and replaced after 30 min, 1 hr, 2 hr, and 3 hr.

The solvent extraction procedure outlined in Chapter 3 was carried out on the heated juices from Experiments 1 and 2. GC analysis, as described in Chapter 3, using a SE54 column was conducted on solvent extracts from all three experiments. Measurements for peak areas of benzaldehyde, benzoic acid, and benzyl alcohol were taken as percentages of the internal standard for Experiments 1 and 2 and as a total area count for Experiment 3. One GC analysis was conducted for Experiments 1 and 3; sufficient juice was available for two replications of the GC analysis for Experiment 2. No statistical analyses were computed because of the lack of replication.

4.3. Results and Discussion

Experiment 1: The effect of temperature on benzaldehyde, benzoic acid, and benzyl alcohol levels in saskatoon juice with seeds removed is depicted in Figure 4.1. At 15°C, the benzaldehyde content of NS juice was similar to that found in earlier studies (chapters two and three) utilising a pulper finisher. Benzaldehyde levels in NS juice remained fairly consistent in the 15-60°C range but tended to decrease very slightly above 65°C. Heat may have oxidized benzaldehyde to benzoic acid (Hatanaka and Kaneda, 1985). The benzoic acid and benzyl alcohol contents of NS juice remained fairly constant at temperatures of 15-85°C.

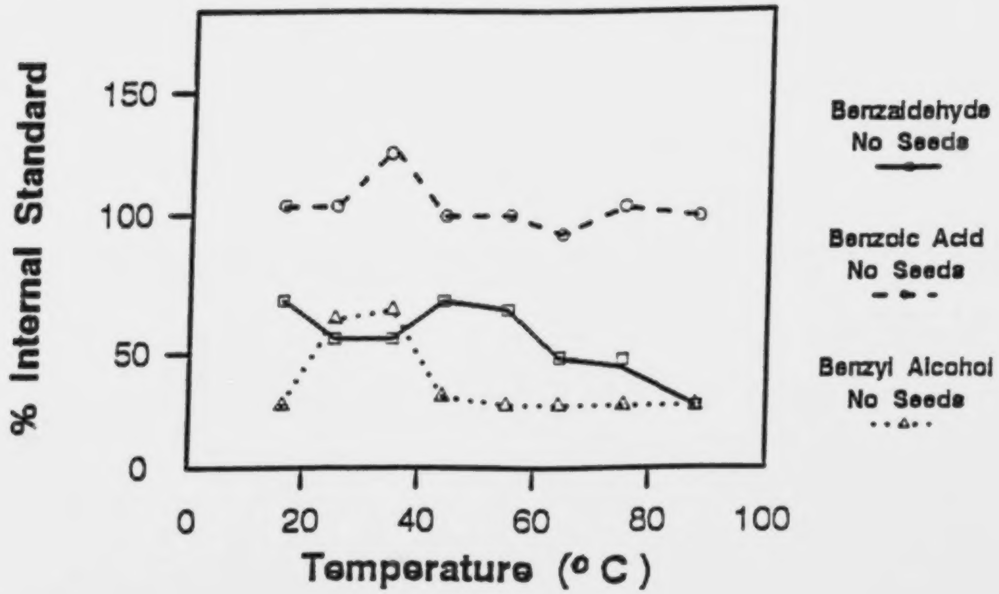

Figure 4.1. Effect of temperature on aromatic compounds in saskatoon juice without seeds.

Experiment 2: The effect of temperature on benzaldehyde, benzoic acid, and benzyl alcohol levels in saskatoon juice with seeds and water is depicted in Figure 4.2. At 20°C the benzaldehyde level of SW juice was 20X higher than NS juice and 30X higher than saskatoon juice with seeds and enzymes determined in a previous study (chapter three). Thus, both seeds and water play a role in the development of benzaldehyde in saskatoon juice. Water assists the enzymes/substrate contact (Whitaker, 1972) which is required for the hydrolysis of amygdalin to benzaldehyde (Wong, 1989). The

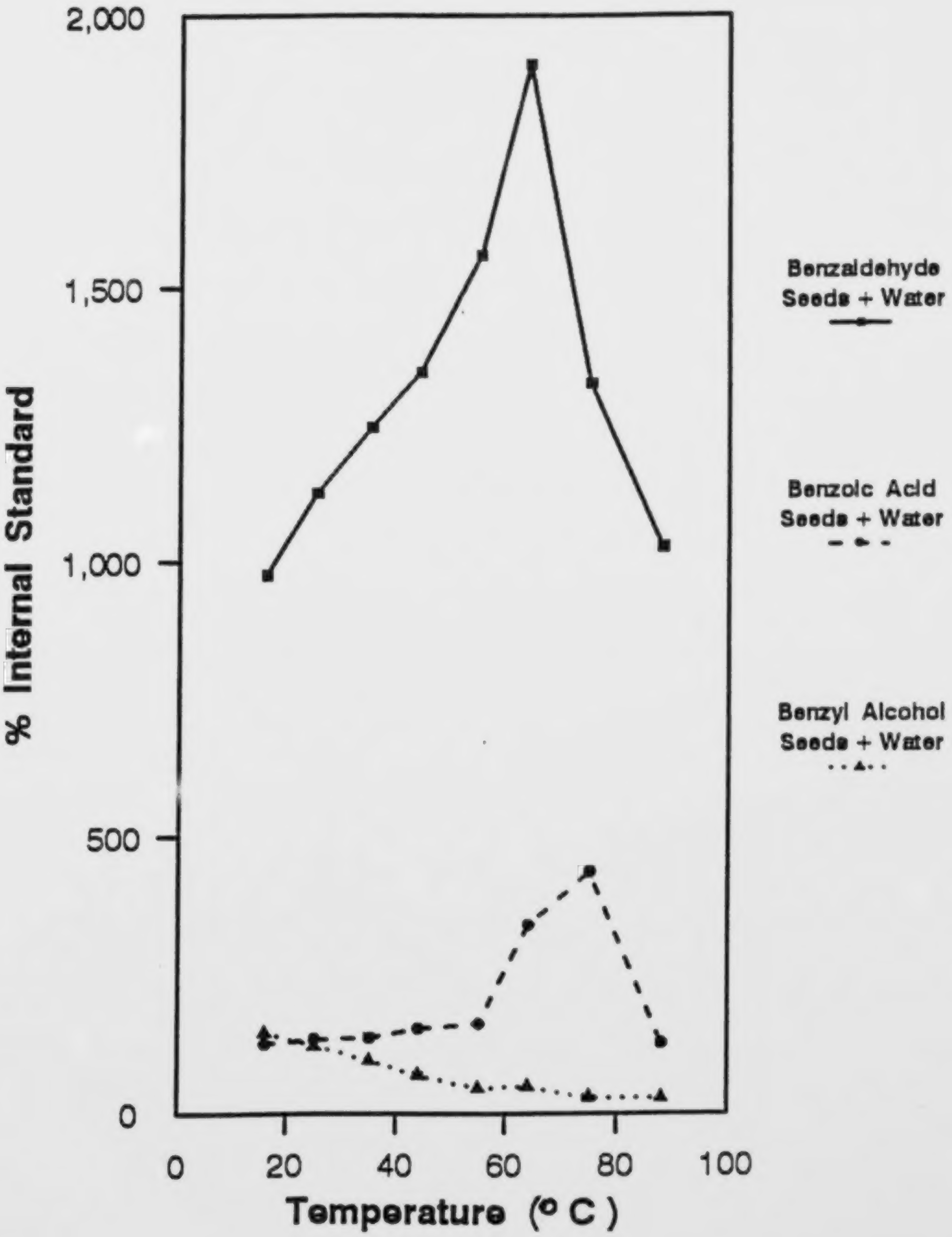

Figure 4.2. Effect of temperature on aromatic compounds in saskatoon juice with seeds and water (SW).

benzaldehyde content of SW juice increased rapidly from 15°C to 65°C and decreased from 65°C to 85°C. This pattern is typical of an enzyme assisted reaction. Swain et al. (1992) demonstrated that two **β**-glucosidases, amgydalin hydrolase and prunasin hydrolase, and mandelonitrile lyase, responsible for the conversion of amygdalin to benzaldehyde in cherries, were present in the cotyledons of cherry seeds. In the present study, the rapid increase in benzaldehyde in SW juice from 15°C to 65°C may be due to the effect of heat on the enzymatic reaction. Heat promotes enzymatic reactions but is also responsible for the denaturation of enzymes at high temperatures (Whitaker, 1972). The reduction of benzaldehyde above 65°C may be due to enzyme denaturation as well as oxidation of benzaldehyde to benzoic acid.

At 20°C the benzoic acid content of SW juice was approximately twice that of NS juice and half that of juice with seeds and enzymes determined in a previous study (chapter three). Thus, the seeds contributed to the benzoic acid content of the juice whereas availability of water did not. The increase in benzoic acid with temperature was similar to that of benzaldehyde but the maximum amount of benzoic acid was attained at a slightly higher temperature (75°C). The high initial and similar, but slightly shifted, pattern of increase in benzoic acid, as compared to benzaldehyde, suggests that benzoic acid may result from oxidation of benzaldehyde. Since benzoic acid is a relatively stable compound, it probably does not readily undergo further chemical reactions in the saskatoon juice. Steam distillation may contribute to the large decrease in benzoic acid found at temperatures above 75°C.

At temperatures of 15°C - 45°C the benzyl alcohol content of SW juice was approximately four times that of NS juice. This suggests that the presence of seeds and/or water promoted release of benzyl alcohol. In apricots, benzyl alcohol was released through enzymatic hydrolysis of glycosidically bound volatiles (Salles et al., 1988). An increase in temperature from 45°C - 85°C resulted in a slight decrease in benzyl alcohol in SW saskatoon juice, suggesting that enzyme systems for benzyl alcohol may differ from those of benzaldehyde and benzoic acid. Steam distillation may account for the decrease in benzyl alcohol at high temperatures.

Experiment 3: The effect of time on the quantities of benzaldehyde, benzoic acid, and benzyl alcohol produced from the distillation of saskatoon berries is shown in Figure 4.3. Steam distillation recovered 96% of the benzaldehyde in the first hour (78% in the first half hour), 3.5% in the second hour, and 0.5% in the third hour. Enzymatic conversion of amygdalin to benzaldehyde was promoted at a rapid rate by the heat and water during the distillation. The dramatic decrease in benzaldehyde production with time may be due to denaturation of the enzyme(s) and/or lack of substrate (Whitaker, 1972). Benzoic acid and benzyl alcohol contents in steam distillates of saskatoon berries followed a more usual recovery pattern with the largest amounts obtained in the first hour and successively less in the following two

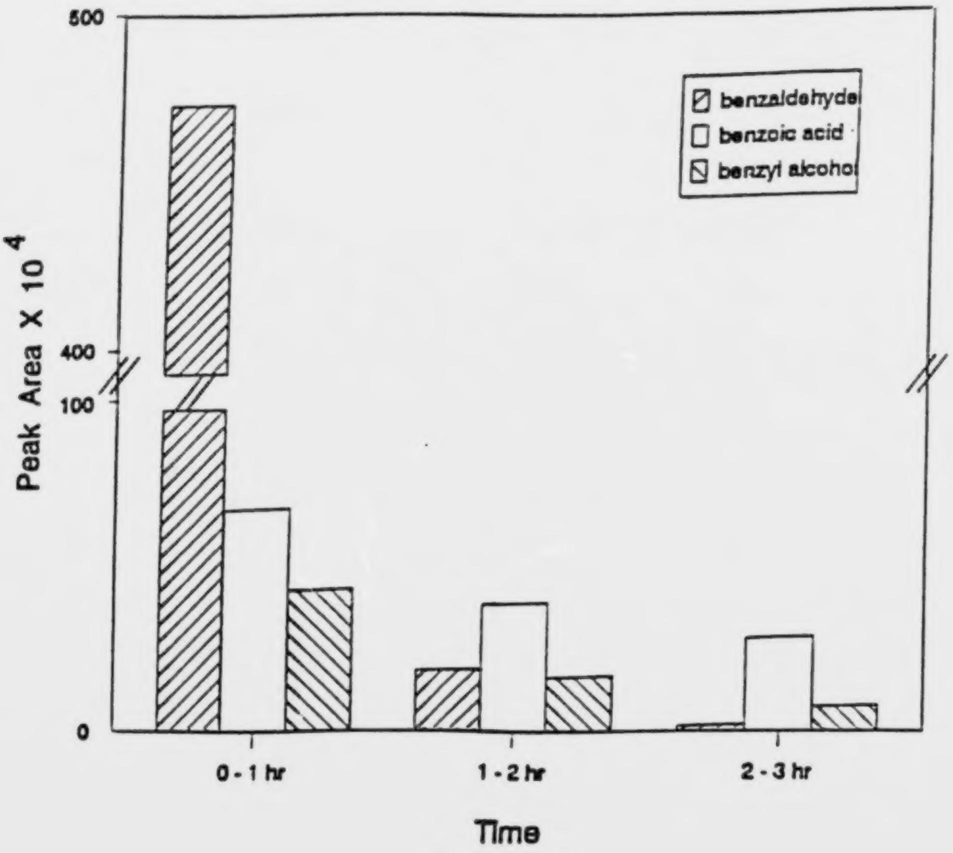

Figure 4.3. Effect of time on aromatic compounds steam distilled from saskatoon berries.

hours as their concentration in the berries decreases. The large initial increase in benzaldehyde did not produce a corresponding large increase in benzoic acid. Benzaldehyde appears to readily steam distill and not oxidize to benzoic acid under these conditions.

The results of the present study demonstrated that small quantities of benzaldehyde, benzoic acid, and benzyl alcohol were present in seedless saskatoon juice; possibly the result of the metabolic pathway which produces these compounds from phenylalanine (Pyysalo, 1976). Tuttas (1977) concluded that benzaldehyde was produced from amygdalin after cells were ruptured and that free benzaldehyde was absent in intact cherry tissues. Other researchers (Honkanen and Pyysalo, 1976; Hirvi et al., 1981; Salles et al., 1988) have determined that benzaldehyde, benzoic acid, and benzyl alcohol are present in significant quantities in fruits such as cloudberries, cranberries, and apricots. The addition of heat and water to berries activates two separate enzyme systems in saskatoon berries which produce benzaldehyde and benzyl alcohol; oxidation of benzaldehyde yields benzoic acid. Thus, heat and water may have a significant impact on the quality of hot processed saskatoon juice. Temperatures up to 60°C promote the production of benzaldehyde which is known to impart sweet almondy characteristics to products. Temperatures above 60°C appear to promote the production of benzoic acid which is not reported to contribute significantly to aroma (Puntari et al., 1985). A further concern, not determined by this study, relates to benzaldehyde stability in hot processed saskatoon juices. During storage and/or with additional processing as in the manufacture of wines, jellies, and syrups benzaldehyde may oxidize to benzoic acid.

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Chapter 5 Quantitative Descriptive Analysis (QDA) of Four Processed Saskatoon Berry Juices

5.1. Introduction

Significant differences in aroma and sugar constituents due to processing treatment have been shown for instrumental data of cold, hot, pectic enzyme, and hemicellulitic + pectic enzyme processed saskatoon berry juices (chapter three). Sensory evaluations of saskatoon berries are unavailable except for observations that homogenized berries smelled warm, sweet, and almondy (Mazza and Hodgins, 1985) and saskatoon jelly had a weak flavour (Mazza, 1979). A complete sensory profile of saskatoon juice, similar to the QDA profiles of peaches (Spencer et al., 1978), grapes (Francis et al., 1992) and apples (Williams and Carter, 1977), is lacking. A detailed profile of the flavour characteristics of saskatoon juice is essential to define quality parameters of the juice. This profile may then be used to determine the characteristics of saskatoon juice processed via different methods and to assist in the interpretation of instrumental data currently available.

This study utilized a QDA panel to establish a sensory profile for saskatoon juice and to evaluate the effect of four processing methods on saskatoon juice quality.

5.2. Materials and Methods

Saskatoon juices from cold (C), pectic enzyme (P), hemicellulitic and pectic enzyme (HP), and hot (H) processes were obtained from experiments conducted in Chapter 3. Briefly, the processing procedures involved: juicing 12 kg lots of thawed saskatoon berries using a pulper finisher at 3°C (C), with the addition of enzymes (P and HP) at 11-20°C, and at 77°C (H); centrifuging to improve juice clarity; and packaging and freezing (-18°C) the juices in 250 mL glass jars 3 mo prior to sensory evaluation. The experiment was replicated four times. For sensory panel training, saskatoon juices were prepared by the same methods on a bench scale using 3 kg lots of berries in a Champion (Model G5NG853S) juicer.

Panelists were selected and trained based on ASTM (1981) QDA procedures with modifications for a small (n=13) initial pool of panelists. During the first two sessions panelists were exposed to a variety of fruit juices (apple, cranapple, applelime, applecot, strawberry, and grape) and developed a large vocabulary list to describe these juices. The following three sessions, each panelist evaluated samples of saskatoon berries (cv. Smoky and wild) as well as laboratory prepared C, HP, and H processed saskatoon juices and created a list of descriptors for the appearance, odour, taste, and oral sensations of the juices. Fifteen appearance, ten odour, three taste, two oral and nine flavour descriptors for saskatoon berries and juice were identified by the panel. In the four subsequent sessions, this lexicon was reduced to twelve common descriptors for saskatoon juice. Six sessions were required to establish definitions for each descriptor using the standards indicated in Table 5.1.

Table 5.1. Lexicon of QDA descriptors and definitions for saskatoon juices.

	DESCRIPTOR	STANDARD (added to 200 g C juice)
APPEARANCE	colour	prune brown - Welch's prune nectar black cherry - canned black cherry juice
	sediment	not sedimenty - centrifuged & filtered sedimenty - uncentrifuged & unfiltered
ODOUR	stale/musty	1.5 mL Quest saskatoon DQ00502
	fresh/fruity	3.0 mL Givaudan apple 10973 0.2 mL Givaudan raspberry 7712B1
	almondy	0.01 mL benzaldehyde
	pruney	60 g juice from frozen berries left uncovered for 1 month in freezer
	sweet odour	2 mL Metarom saskatoon MC2612
	medicinal	1 mL benzyl alcohol
TASTE	sweet	4 g sucrose
	sour	0.4 mL 50 % citric acid
ORAL	dry	10 mL 1 % tannic acid
FLAVOUR	strength	weak - 20 g water

To select panelists, a series of 23 triangle tests was conducted to evaluate each panelist's ability to discriminate among saskatoon juices which differed in appearance, odour, taste, oral and flavour attributes. For each descriptor, except colour, two triangle tests were conducted with the addition of the standards to saskatoon juice at the level indicated in Table 5.1 and then at reduced levels. Eight panelists (4M + 4F, aged 25-40) were selected based on completion of the triangle test series with > 68% correct responses.

During further training, the judges evaluated lab samples of C, P, and H juices, and developed a QDA scorecard consisting of eleven 15 cm line scales. The scales were anchored at 0 and 15 with the following terms: prune brown colour and black cherry colour; clear and cloudy/sedimenty; stale/musty and fresh/fruity; not almondy and very almondy; not pruney and very pruney; not sweet (odour) and very sweet (odour); not medicinal and very medicinal; not sweet (taste) and very sweet (taste); not sour and very sour; not dry and very dry; and weak flavour and strong flavour.

Procedures were developed for evaluation of samples. For evaluation, frozen juices were thawed (1 hr in 20°C water) and poured in 20 mL aliquots into 25 mL test tubes with screw tops. One set of juice samples was prepared for evaluation of appearance. Samples were evaluated for colour and clarity against a white countertop and wall in a room with artificial lighting. A second set of juices in test tubes was presented, at 20°C, to each panelist in a sensory evaluation booth with red lighting. Panelists were directed to remove the test tube lid and take three sniffs to evaluate juice odour. Judges then poured each juice sample into 50 mL styrofoam cups, tasted, swallowed and scored the remaining descriptors. Panelists were provided with 20°C water to rinse between samples.

Judges were evaluated for their ability to discriminate among samples and their reproducibility by conducting four panels, on different days, using four samples of saskatoon juice (C, P, and 2 H) which were prepared in the lab. Scores for each of the eleven descriptors were measured to the nearest 0.1 cm on the 15 cm scales. Analysis of variance (ANOVA) of the data showed panelists could discriminate among the samples with respect to colour, clarity, musty/fresh, almondy, pruney, sweet odour, medicinal, and sour. Panelists were consistent in their responses to sweet taste, dryness, and flavour strength but these descriptors did not vary significantly among the samples. ANOVA of replicate judgements by panelists showed no significant differences for each of the eleven descriptors evaluated. Not all eight panelists were able to discriminate among the samples and replicate their responses for each individual characteristic. Thus, for the study, six of the eight panelists were chosen for each characteristic based on their ability to discriminate among the four saskatoon juices (ie. colour data were collected from judges 1, 2, 4, 5, 6, and 7 while almondy data were collected from judges 1, 2, 3, 4, 5, and 6.)

At each experimental session, each panelist received one sample from each of the four treatments (C, P, HP, and H) in random order. Since the yield of H juice was high enough, a duplicate H sample was included at every other session to monitor panel reproducibility. Sessions were held at 11 am and 2 pm daily for six days. Juices were presented and scored as per the training sessions. Samples from each of the four replications of the four treatments were evaluated in triplicate.

Data were subjected to analysis of variance. Student-Neuman-Keuls (SNK) multiple range test (O'Mahony, 1986) was used to determine significant differences among treatment means for each of the eleven characteristics. Sources of variation were: processing treatments (t=4), replications (r=4), panelists (p=6), and panel sessions (s=4). ANOVA and SNK were also used to evaluate the consistency of the panel with respect to the duplicate H juices.

5.3. Results and Discussion

Mean sensory scores for cold (C), pectinase (P), hemicellulase and pectinase (HP), and hot (H) saskatoon juices are presented in Table 5.2. No significant differences were found among duplicate H samples. Thus mean scores for duplicate H juices are not included.

H juice was significantly ($p \leq 0.001$) more black cherry coloured than HP, P, and C juices. C juice was significantly more prune brown coloured than HP juice. P juice was similar in prune brown colour to both C and HP juices. The increased prune brown colour observed in C, P, and HP juices corresponds to the increase in yellowness and lightness determined instrumentally (chapter three). Crushed fruit browns due to oxidative enzyme reactions (Moyer and Aitken, 1980). Heat processing inactivates the oxidative enzymes to prevent browning (Dimick et al., 1951) and, for fruits containing anthocyanin pigments,

Table 5.2. Means[1] and standard errors for sensory analyses of processed saskatoon berry juices.

Attribute	Processed Juice Treatment				SEM[2]
	Cold C	Enzyme P	Enzyme HP	Hot H	
APPEARANCE					
Colour	4.63^{c}	5.64^{bc}	6.83^{b}	12.71^{a}	0.44^{***}
Clarity	9.73^{a}	7.98^{a}	6.51^{a}	3.21^{b}	0.90^{**}
ODOUR					
Musty/Fresh	4.33^{b}	4.81^{b}	4.98^{b}	11.85^{a}	0.24^{***}
Almondy	3.90^{c}	5.50^{b}	5.58^{b}	11.80^{a}	0.25^{***}
Pruney	10.44^{a}	9.54^{a}	9.55^{a}	3.83^{b}	0.26^{***}
Sweet Odour	5.10^{c}	6.31^{b}	6.98^{b}	10.23^{a}	0.26^{***}
Medicinal	5.94^{b}	9.57^{a}	9.35^{a}	2.77^{c}	0.28^{***}
TASTE					
Sweet	5.85^{b}	7.10^{b}	7.25^{b}	10.24^{a}	0.41^{***}
Sour	7.14^{a}	7.02^{a}	6.77^{a}	4.37^{b}	0.36^{***}
ORAL					
Dry	8.71	9.03	8.68	9.05	0.20^{ns}
FLAVOUR					
Strength	8.51^{b}	9.68^{a}	9.37^{a}	10.13^{a}	0.25^{**}

[1] Sensory means are the averages of 72 determinations by 6 trained panelists

[2] Standard error of the mean

[ns] Not significant

[abc] Means within the same row sharing a common letter are not significantly different at ** = $p \leq 0.01$ and *** = $p \leq 0.001$

results in deeper purple/red colours in heated as compared to unheated juices (Tressler et al., 1980).

The H juice had significantly ($p \leq 0.01$) greater clarity than HP, P, and C juices which were similar. The H treatment used in the present study was based on one for blueberries (Yang, 1984) which also reported increased clarity for H versus C blueberry juice.

The H juice, which was characterised as being fresh/fruity, differed significantly ($p \leq 0.001$) from C, P, and HP juices which were musty/stale. Of the aroma constituents identified in saskatoon juices (chapter three) only linalool oxide is reported to be musty (MacLeod and Pieris, 1983). Levels of linalool oxide were higher in C, P, and HP juices than in H juice but the differences were not significant (chapter three). Instrumental analysis determined a significant increase in butyl butanoate in H juice over C, P, and HP juices (chapter three). This ester is characterised as being fresh, sweet, and fruity (Flath and Forrey, 1977) and has an important role in pear aroma (Chua et al., 1987). Thus, butyl butanoate may contribute to the fresh/fruity notes in H juice. The intense aroma of butyl butanoate, along with increases in other volatiles in H juice, may mask musty notes. The masking potential of butyl butanoate may explain why C, P, and HP juices were judged more musty than H juice although the linalool oxide content of the juices was not significantly different.

The H juice had significantly ($p \leq 0.001$) more almondy odour than HP and P juices which were alike and higher in almondy odour than C juice. These data support the increases found in the benzaldehyde content of juices (chapter three). Benzaldehyde has characteristically sweet almondy notes (Dravnieks, 1985). Benzaldehyde appears to be a fairly strong aroma compound judging by the ability of the panelists to detect the slight increase in benzaldehyde from C to P juices. Increases in temperature have been shown to promote enzymatic conversion of amygdalin to benzaldehye (Schmid and Grosche, 1986). The dramatic increase in benzaldehyde in H juice, together with its strength as an aroma compound, resulted in a sweet almondy characteristic which may have masked or enhanced other sensory characteristics.

The H juice was significantly less pruney in odour than C, P, and HP juices which were similar. No volatile constituents were found instrumentally which are known to have a pruney note (chapter three). The difference in perception of pruney notes in H versus the other juices may result from an absence of a particular volatile or a masking effect from the fresh/fruit and sweet/almondy notes found in H juice.

The H juice had a significantly ($p \leq 0.001$) stronger sweet odour than HP and H juices which were similar and also stronger in sweet odour than C juice. Benzaldehyde is known to contribute a sweet odour as well as almondy notes to products. Panelist's perception of sweet odour in the four juices appears to follow the same pattern, and order of magnitude, as that for almondy. Thus, differences in sweet odour among the saskatoon juices may be due to differences in benzaldehyde content.

The P and HP juices were characterised as significantly ($p \leq 0.001$) more medicinal than C juice which was also more medicinal than H juice. Benzyl alcohol was used by the trained panel as a standard to define medicinal. Instrumental analysis showed that there was significantly ($p \leq 0.001$) more benzyl alcohol (2X and 5X) in P and HP juices than H and C juices, respectively (chapter three). The presence of benzyl alcohol may account for the stronger medicinal notes in HP and P juices than in C juice. Benzyl alcohol was also determined in H juice which was not perceived by the juices as very medicinal. The high level of benzaldehyde which provides a strong sweet almondy aroma may mask the medicinal notes in H juice.

The H juice was scored significantly ($p \leq 0.001$) sweeter than HP, P, and C juices which were similar. Instrumental analysis determined that H juice contained significantly ($p \leq 0.05$) higher quantities (approximately 1%) of sugars than HP, P, and C juices (chapter three). Thus the sensory results for sweetness support the analytical sugar findings.

The C, P, and HP juices were similar and significantly ($p \leq 0.001$) more sour than H juice. Instrumental analyses of the major acids (quinic, malic, and citric) present in saskatoon juice did not determine any significant differences in the quantities of the three acids among the four saskatoon juices (chapter three). The perceived sourness of C, P, and HP juices may be due to the lower level of sugars determined in these juices compared to H juice.

All four saskatoon juices were judged to be similar in dryness. The scores for dryness were consistently just above 8.5 cm on the 15 cm scale. During training, panelists scored the dryness of cranberry juice as 15 cm. The quantity of quinic acid, which contributes to dryness, in saskatoon juice was calculated to be approximately a half to a third that of cranberry juice (Coppola and Starr, 1986). The dryness scores given to saskatoon juices suggest that quinic acid may be responsible for the perception of this attribute.

The H, HP, and P juices were similar and significantly ($p \leq 0.01$) stronger in flavour than C juice. The strong fresh/fruity, almondy, sweet odour, and sweet taste of H juice may account for its overall stronger flavour than that of C juice. The strong medicinal odour in HP and P juices may have contributed to a resulting stronger flavour than found in C juice.

This study proposed a lexicon of eleven descriptors for processed saskatoon juice including: colour (prune brown to black cherry), clarity, freshness (musty/stale to fresh/fruity), almondy, sweet odour, pruney, medicinal, sweet taste, sour, dry, and flavour strength. Findings of this study indicate that the method of processing saskatoon juice affected the sensory characteristics of the juice. Cold processed juices were characterised as prune brown coloured, cloudy, musty/stale, pruney, sour, dry, and weak flavoured. Enzyme (P and HP) juices were described as prune brown coloured, cloudy, musty/stale, slightly almondy, pruney, medicinal, sour, and dry. Hot processed juices were characterised as black cherry coloured, clear, fresh/fruity, almondy, sweet (in odour and taste), and dry. Many relationships between sensory and instrumental data (chapter three) were suggested including: colour with HunterLab 'L' and 'a' values; musty/stale with linalool oxide; fresh/fruity with butyl butanoate; almondy with benzaldehyde; sweet odour with benzaldehyde; medicinal with benzyl alcohol; sweet taste with sugars; and dry oral sensation with quinic acid. Future research utilising multivariate techniques is required to determine correlations between sensory and instrumental data. This study focused on the use of a trained panel to determine differences among the four processed saskatoon juices. Research using consumer panels is necessary to determine the effect of the differences among processed juice treatments, detected by trained judges, on consumer acceptance.

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Chapter 6 Free-Choice Profiling (FCP) of Four Processed Saskatoon Berry Juices

6.1. Introduction

Saskatoon juices processed by cold, enzyme, and hot processing methods have been shown : 1) instrumentally to have different levels of colour, sugar and volatile constituents (chapter three); and 2) sensorially, by a QDA panel, to differ in colour and clarity, musty, fruity, almondy, sweet, pruney, and medicinal odour, sweet and sour taste, as well as flavour strength (chapter five). However, the degree to which the sensory differences among the processed juices, detected by the trained judges, may be perceived by consumers has not been determined. Information on consumer acceptance of the different juice treatments would also be invaluable to processors.

Free-choice profiling (FCP) (Williams and Langron, 1984) is a relatively new sensory technique which permits untrained panelists to develop individual vocabularies to describe and differentiate among products. Generalised Procrustes analysis (GPA) is used to statistically analyse differences among products based on data from individual panelist's vocabularies rather than means from group data (Gower, 1975). As the panelists are untrained hedonic differences among products may be determined using analysis of variance.

FCP has been employed successfully in naive consumer differentiations of whisky (Guy et al., 1983) and meat (Steenkamp and van Trijp, 1987). FCP was not successful in describing differences in fish due to spoilage (Quarmby and Ratkowsky, 1988); consumer inability to develop a vocabulary was cited as a major problem. Thomson's review (1989) indicated that FCP has potential for use with naive consumers but further studies are required. Thus, *consumer panel studies of processed saskatoon* juices utilising FCP as compared to QDA (chapter five) are warranted to provide a further evaluation of the FCP technique.

The present study was conducted to evaluate differences in sensory characteristics and hedonics among four processed saskatoon juices as determined by untrained consumer panelists using FCP. Data from FCP panels were also compared to existing trained QDA panel data.

6.2. Materials and Methods

Saskatoon juices from cold (C), pectic enzyme (P), hemicellulitic and pectic enzymes (HP), and hot (H) processes were obtained from experiments in Chapter 3. A pulper finisher was used to press 12 kg lots of fruit for all treatments. The juices were centrifuged, packaged in 250 mL glass jars, and stored frozen (-18°C) for 3 mo prior to sensory evaluation. The experiment was replicated four times. Laboratory samples of each treatment were prepared for panel training according to the methods indicated in Chapter 3, except the juice was pressed in 3 kg lots in a Champion (Model G5NG853S) juicer.

Two consumer FCP panels were conducted; the first at the Food Quality Branch (Alberta Agriculture, Food and Rural Development) (n=24), and the second at the Department of Food Science and Nutrition (University of Alberta) (n=24). At each location participants were divided into two groups with 10-14 members. The first panel was conducted at the same time as the QDA panel; the second panel followed 6 mo later. A total of 32 women and 16 men, aged 20-60 yr, participated in the study. Saskatoon berries were familiar to 37 panelists but were new to the other 11.

On day one, each group was given a brief explanation of the purpose of the study and three samples of laboratory prepared C, P, and H juices in 125 mL white styrofoam cups. Panelists developed individual vocabularies to describe the three juices according to appearance, odour, taste, and oral sensations. Anchor terms to describe the lower and upper limits of each vocabulary term were also created. The vocabulary and anchor terms were reviewed with each panelist to insure that each scale was unidirectional and not hedonic in nature. An individual scorecard with 15 cm line scales, anchored with each individual's unique set of terms, was created for each panelist. A 15 cm hedonic line scale, anchored with 'dislike' and 'like', was added at the end of each scorecard. Four scorecards were copied for each panelist and coded to correspond with four 125 mL white styrofoam cups.

On day two, one 250 mL jar from each replicate of each of the four treatments was thawed (1 hr in 20°C water). The four replicates of each treatment were combined, due to limited quantities, to provide 1 L of juice per treatment. Samples of approximately 30 mL aliquots of each treatment were poured into coded 125 mL styrofoam cups for serving to the judges. Each panelist was presented with four individual scorecards and a glass of water for rinsing between samples. One of the four treatments was presented and evaluated. The sample and scorecard were removed before serving the next sample to the panelist. All four treatments were presented to panelists in a random order within the group.

A QDA panel was trained according to ASTM (1981) procedures during 33 panel sessions as described in Chapter 5. Six panelists evaluated eleven juice attributes using 15 cm line scales anchored at 0 and 15 with the following terms: prune brown colour and black cherry colour; clear and cloudy/ sedimenty; stale/musty and fresh/fruity; not almondy and very almondy; not pruney and very pruney; not sweet (odour) and very sweet (odour); not medicinal and very medicinal; not sweet (taste) and very sweet (taste); not sour and very sour; not dry and very dry; and weak flavour and strong flavour. Panelists evaluated randomly presented samples from each of the four replications of the four treatments in triplicate.

Line scales from FCP and QDA panels were measured to the nearest 0.1 cm. Due to the varying number of characteristics used by consumer panelists, data matrices were padded with zeros as required. Each QDA panelist's data were averaged for each treatment (four replicates of each treatment evaluated in triplicate). Data from the each of the consumer panels and from the QDA panel were analysed separately by GPA using OP&P's Procrustes-PC version 2.2 (Dijksterhuis and van Buuren, 1989) to obtain consensus configurations. Similar vocabulary terms from both FCP panels were grouped and hand plotted as areas on GPA consensus configuration plots. Hedonic data from the two consumer panels were subjected to analysis of variance (ANOVA). Sources of variation were: processing treatments (t=4), panel sessions (s=2), and panelists (p=24). Significant differences ($p \leq 0.05$) among hedonic responses were identified by Student-Newman Keul's multiple range test (O'Mahony, 1986).

6.3. Results and Discussion

The GPA consensus configurations for the consumer and trained panels are presented in Figure 6.1. Each of the three panels gave similar consensus configurations. The differences among the four treatments are represented on the GPA plot by their distance from the 0,0 point, the location which indicates no difference. Principal coordinate one (PC1) shows a large difference between H and C, P, and HP juices which were similar. Principal coordinate two (PC2) separates C from H juices, and H from P and HP juices which were similar.

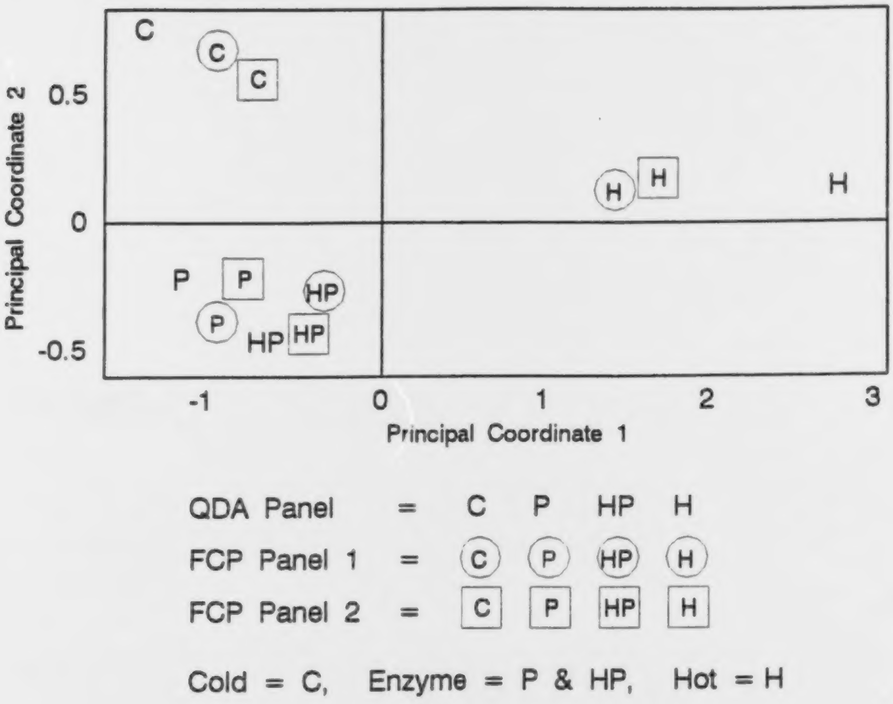

Figure 6.1. Generalised Procrustes consensus configurations for FCP and QDA panels for processed saskatoon berry juices.

For the QDA panel, the contribution of each juice treatment to the overall variation among the treatments is presented in Table 6.1. Hot juice contributed the greatest amount of variation, followed by C juice, and then by P and HP juices, which were similar. The QDA consensus configuration with two principal coordinates accounted for 95.2% of the variation among treatments. Only 3.5% of the variation was residual; thus, a third principal coordinate would not significantly improve interpretation of the data.

Table 6.1. Generalised Procrustes analysis of variance of QDA consensus configurations for processed saskatoon berry juices.

Treatment	Percentage Variation		
	Consensus	Residual	Total
Cold (C)	17.1	1.4	18.5
Enzyme (P)	7.5	1.0	8.5
Enzyme (HP)	6.0	0.7	6.7
Hot (H)	64.6	0.4	65.0
Total	95.2	3.5	98.7

The GPA of variance of principal coordinates for the QDA and FCP panels is given in Table 6.2. The data show that the two principal coordinates accounted for a greater percentage of the variation among treatments for the QDA panel (95.2%) than for the FCP panels (83.6-82%). As expected, the QDA panel determined greater differences among the juices according to the two principal coordinates than the FCP panels. The QDA panel showed a greater percentage of the variation among treatments attributable to PC1 than the two FCP panels which were similar. FCP panels showed similar percentages of variation among the juices attributable to PC2; these PC2 variations were greater than that of the QDA panel.

Table 6.2. Generalised Procrustes analysis of variance of principal coordinates for QDA and FCP panels.

Panel Type	Percentage Variation		
	Principal Coordinate 1	Principal Coordinate 2	Total
QDA	87.5	7.7	95.2
FCP 1	68.7	14.9	83.6
FCP 2	67.9	14.1	82.0

The terms which the consumer panelists (FCP) used to describe the saskatoon juices are presented in Table 6.3. Terms which appeared to describe the same stimulus were grouped together and the frequency of their use enumerated according to the group. The percentages of FCP panelists using descriptors similar to those of QDA panelists were : fresh/fruity, 100%; colour, 98%; sweet taste, 85%; clarity, 71%; sour, 48%; almondy, 42%; flavour strength, 42%; dry, 35%; medicinal, 29%; pruney, 27%; musty, 25%; and sweet odour, 13%. Thus, all eleven QDA descriptors were readily perceived by consumer panelists with fruitiness, colour, sweetness, and clarity being important to the majority of them.

Table 6.3. FCP (n=48) descriptors for processed saskatoon berry juices

	MOST COMMON	OTHERS	NO. OF PANELISTS
APPEARANCE	cherry/wine red	purple, plum, grape	47 ^
	brown	dark brown, dark	15 ^
	sediment/cloudy	particles, grainy, residue, pulpy, opague, turbid, filthy, murky	34 ^
	shiny	glossy, bright	9
ODOUR	fruity		39 ^
	almondy	marashino cherry	20 ^
	medicinal	alcoholic, hospital, hand lotion	14 ^
	pruney		13 ^
	musty	earthy, woody, old potato	12 ^
	berry		10
	fresh		9 ^
	sweet		6
TASTE	sweet		41 ^
	sour	tart, acidic	23
	bitter		10
ORAL	astringent	dry	17
FLAVOUR	strength		20

^ indicates descriptors which differentiated among the four juice treatments.

The FCP panels determined 17 descriptor groupings, seven of which were similar among the four juice treatments. For the ten terms which differentiated among the treatments, data for both FCP panels were combined and plotted onto the consensus configuration (Figure 6.2). The results indicated that C juice was characterised as brownish, sedimenty, musty/earthy, and pruney; P and HP juices were brownish, sedimenty, musty/earthy, pruney, and medicinal/alcoholic; H juice was wine red, clear, fresh/fruity, almondy, and sweet. These results agree with those of the QDA panel (chapter five).

The QDA panel determined differences in sweet odour and sourness among the juices which were not detected by the FCP panels. In the case of sweet odour, insufficient FCP data (n=6) were available to accurately determine differences among treatments. Sourness had sufficient data but differences among juice treatments were not observed. The lack of differentiation by the FCP panels for sourness may be due to the similarity of the samples for this attribute.

The hedonic means , for the degree of like and dislike, for the treatments were: H, 10.3; HP, 6.7; C, 6.0; and P, 5.1. out of 15. All the juices differed significantly ($p \leq 0.05$). The low hedonic score of P juice is probably due to the medicinal note. Although HP juice also had a medicinal note it contained three times more benzaldehyde than C or P juices (chapter three)

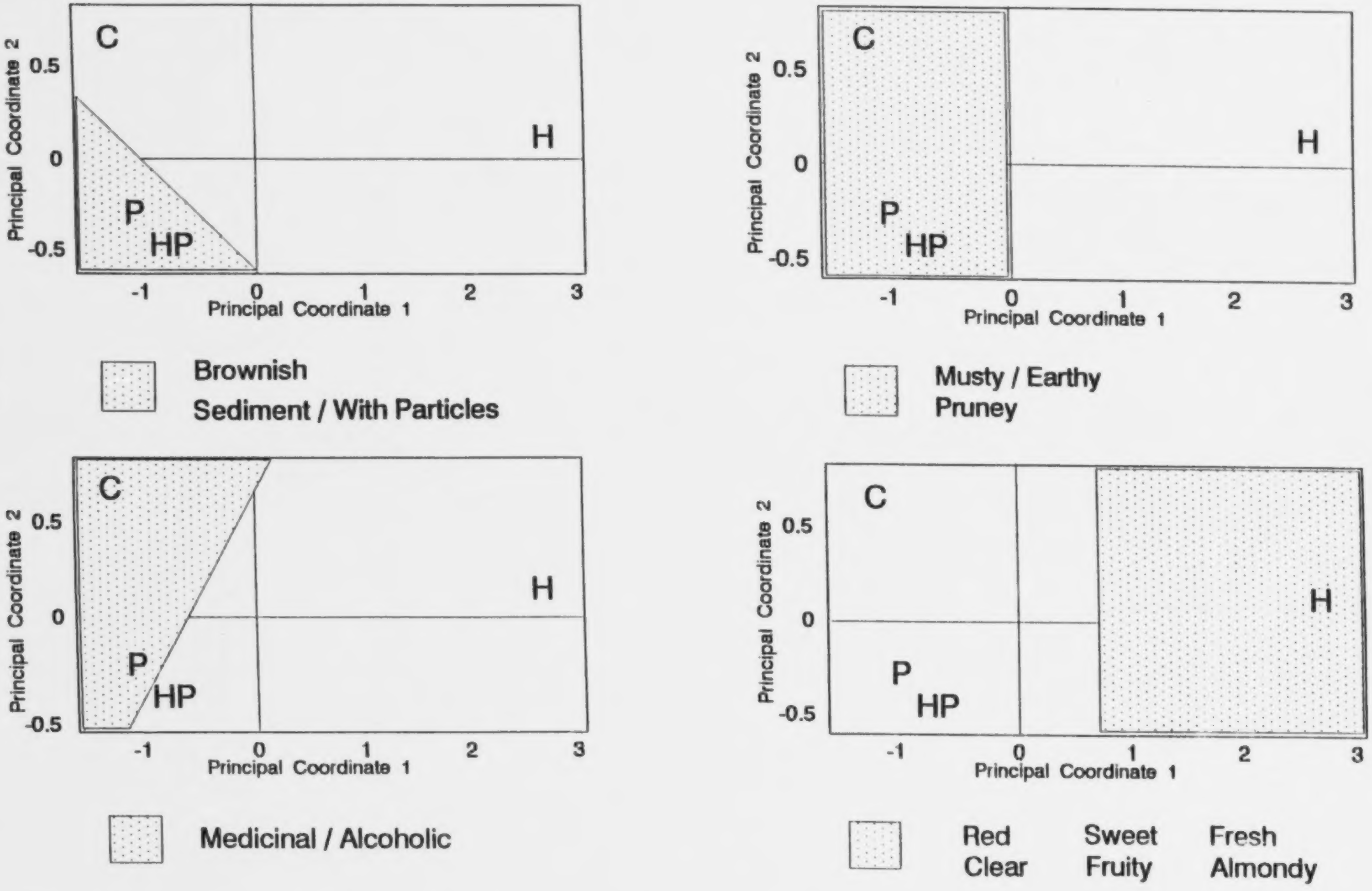

Figure 6.2. Location of free-choice profiling descriptors for C, P, HP, and H processed saskatoon berry juices.

which gave it an almondy note. The almondy note may have contributed to the higher hedonic score given to HP juice than to C juice. The high score given to H juice may be due to the large differences in colour, clarity, sweetness, fresh fruitiness, and almondiness as compared to HP, C, and P juices.

The results of this study showed that consumer panelists, using FCP, were able to differentiate among four saskatoon juice treatments with respect to colour, clarity, musty, pruney, medicinal, sweet, fruity, fresh, and almondy characteristics. GPA demonstrated that results from FCP panel corresponded well to trained QDA panel data. As expected, the degree of differentiation among the four juices was greater for the QDA panel than the FCP panels. FCP with naive consumer panelists was successful in describing and quantifying ten sensory differences among the juices; but was not able to determine two small sensory differences in the juices, sweet odour and sourness, which were perceived by a trained QDA panel. Results from the FCP panels were obtained in five days versus 3 mo for the QDA panel study. Hedonic data from the FCP consumer panels indicated that H juice was liked more than C, P, and HP juices.

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Chapter 7 Generalised Procrustes Analysis of Instrumental and Sensory Data of Four Saskatoon Berry Juices

7.1. Introduction

Instrumental data for colour, sugars, acids, and volatile constituents of four processed saskatoon juices are available (chapter three). Results for eleven sensory attributes for the same four processed saskatoon juices have been determined by a QDA panel (chapter five). Significant differences among the juices were found both instrumentally and sensorially. However, relationships between instrumental and sensory data for the juices have not been established.

Generalised Procrustes analysis (GPA), a statistical technique, has been used to interpret differences among products based on individual data sets (Gower, 1975). Free-choice profiling (FCP) allows each panelist to develop a unique vocabulary to describe differences among products (Williams and Langron, 1984). By comparing differences among the products according to each panelist's vocabulary terms, GPA (through the processes of translation, rotation, and scaling) establishes relationships among the terms from each of the individual vocabularies (McEwan and Hallett, 1990). GPA was used successfully with QDA and FCP panel data to determine sensory differences among four processed saskatoon juices (chapter six).

Liardon et al. (1984) compared GC analytical data and QDA sensory data of fifteen coffees by two separate GPA. The analytical and sensory configurations which were superimposed on each other did not reveal relationships between the sensory descriptors and specific GC components. The authors concluded that coffee descriptors were probably linked to combinations of GC components. Other published reports using GPA to relate sensory and analytical data are lacking.

The sensory and instrumental data available (chapters five and three, respectively) have both measured differences among the same four juices; thus it should be possible to analyse both data sets together in one GPA. Combining the sensory data for the juices with the unidentified GC peak data may also focus the GC peak identification efforts on those volatile components which correlate to sensory descriptors.

This study was conducted to evaluate the use of GPA to establish instrumental and sensory relationships among four processed saskatoon juices.

7.2. Materials and Methods

Saskatoon juice data for instrumental and sensory relationships were obtained from Chapters 3 and 5, respectively. These data determined differences among saskatoon berries which were juiced, in 12 kg lots, by cold (C), pectic enzyme (P), hemicellulitic and pectic (HP), and hot (H) methods using a pulper finisher. Data were recorded for all four replicates of the experiment. Instrumental measurements were obtained for: HunterLab colour -

'L' 'a' and 'b' values; HPLC sugars; HPLC acids - quinic, malic, citric, ascorbic, and fumaric; and GC volatiles of solvent extracts of juices - 30 peaks using an SE54 column, and 23 peaks using a Supelcowax 10 column. Colour, sugar, and acid determinations were made in duplicate. Solvent extractions for GC volatile analysis were done in quadruplicate. A sensory QDA panel, trained during 33 sessions according to ASTM (1981) procedures, evaluated: appearance including colour, and clarity; odour including musty, fresh fruity, almondy, pruney, sweet, and medicinal; taste including sweet and sour; oral sensation including dryness; and flavour strength of the four saskatoon juice treatments. Sensory data were collected in triplicate for each of the four replicates of the four treatments.

For each treatment, all replicate instrumental and sensory data were averaged to give one value for each instrumental and sensory measurement. Three data matrices were created containing: 1) colour, sugar, acid, and GC SE54 column peak data; 2) colour, sugar, acid, and GC Supelcowax 10 column peak data; and 3) QDA sensory data. OP&P's Procrustes-PC version 2.2 software (Dijksterhuis and van Buuren, 1989) was used for two GPA. The first analysis correlated matrices 1 and 3 and the second analysis correlated matrices 2 and 3. A consensus configuration describing the relationships among the four saskatoon juices was plotted. GPA instrumental and sensory results were grouped and plotted according to appearance, taste and oral sensation, and odour and flavour strength. Unidentified GC peaks, which related to sensory characteristics on the GPA correlation plots, were tentatively identified (HP #041 database as in chapter three) where possible.

7.3. Results and Discussion

The consensus configurations for the two GPA analyses, one including GC SE54 column data and the other including GC Supelcowax 10 column data, were the same. A consensus configuration depicting the differences among the C, P, HP, and H juices treatments is shown in Figure 7.1. C juice is situated in the upper left quadrant, P and HP juices are in the lower left quadrant, and H juice is located to the far right of the consensus plot. Subsequent correlation plots for appearance, taste, and odour and flavour strength are based on the same two principal coordinates (PC1 & PC2) but their scales differ. Since the correlation plots relate each instrumental or sensory data set to the two principal coordinates (PC1 & PC2) numerical values for instrumental and sensory correlations are not available.

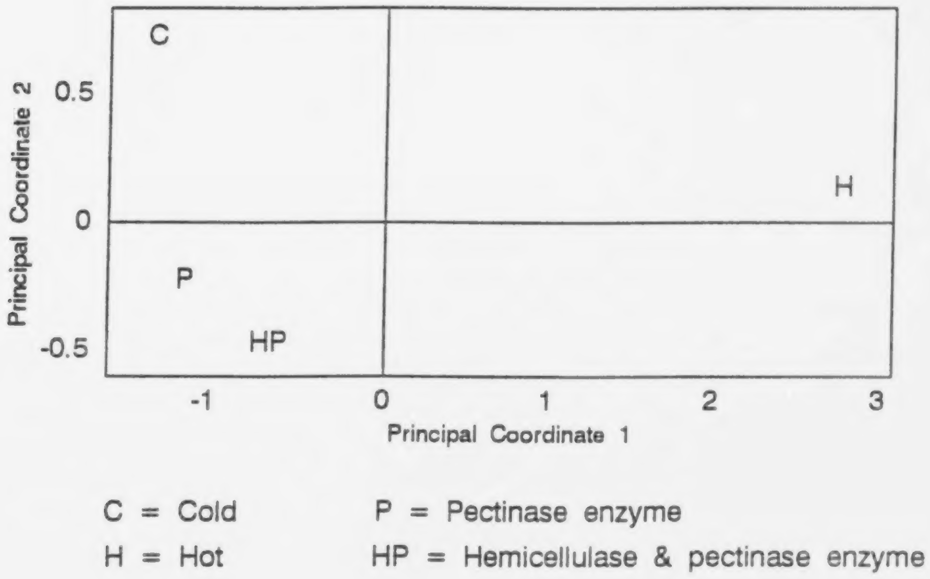

Figure 7.1. GPA consensus plot of processed saskatoon berry juices.

Both GPA correlation plots (based on SE54 and Supelcowax 10 data) for appearance were similar; one is depicted in Figure 7.2. The plot of appearance includes HunterLab 'L', 'a', and 'b' values as well as QDA colour and clarity. The prune brown sensory descriptor correlated well with HunterLab 'L' and 'b' values for lightness and yellowness, respectively. The Hunterlab 'L' and 'a' values for darkness and redness, respectively, related to the positive end of PC1 along with the sensory dark cherry colour. Sensory clarity data were plotted but instrumental data were not available to establish correlations.

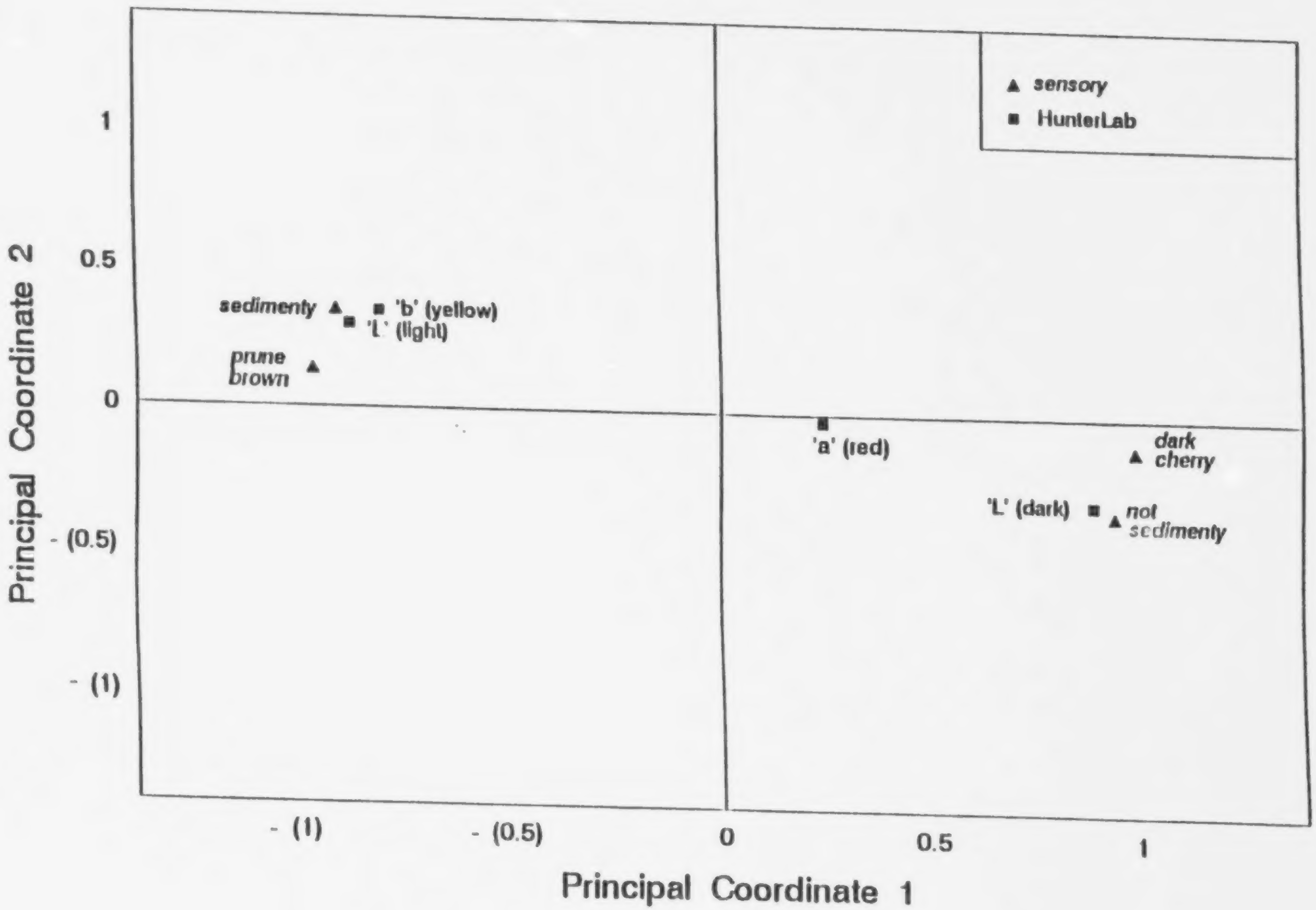

Figure 7.2. GPA correlation plot of instrumental and sensory appearance of processed saskatoon berry juice.

The two GPA correlation plots for taste were similar; one is shown in Figure 7.3. The plot of taste includes data for HPLC sugars and acids together with QDA sweet, sour, and dry sensory data. Sweet taste and sugars are both located at the positive end of PC1. No relationship between sour taste and acids was established. Significant differences were not determined among the four treatments with respect to acid contents (chapter three). Sour and sweet are situated at opposite ends of PC1. C, P, and HP juices had a lower ratio of sugar to acid as compared to H juice. This resulted in a perception of increased sourness in C, P, and HP juices and/or increased sweetness in H juice. The GPA correlation plot indicates quinic, citric, and ascorbic acids may be correlated with dryness. Quinic acid contributes to dryness in cranberries (Coppola and Starr, 1986) and may be the main contributor to dryness in saskatoon juices.

GPA correlation plots for GC instrumental data and sensory odour as well as flavour strength data are shown in Figures 7.4 (SE 54 column) and 7.5 (Supelcowax 10 column). Both plots show a strong relationship between almondy and benzaldehyde along the positive end of PC1. Benzaldehyde imparts almondy and sweet odours (Dravnieks, 1985). This strong relationship between benzaldehyde and sweet odour is also evident on both GPA odour and flavour strength plots. Phenyl ethanol, which has been described as floral and sweet (Dravnieks, 1985), also appears in the same quadrant as sweet

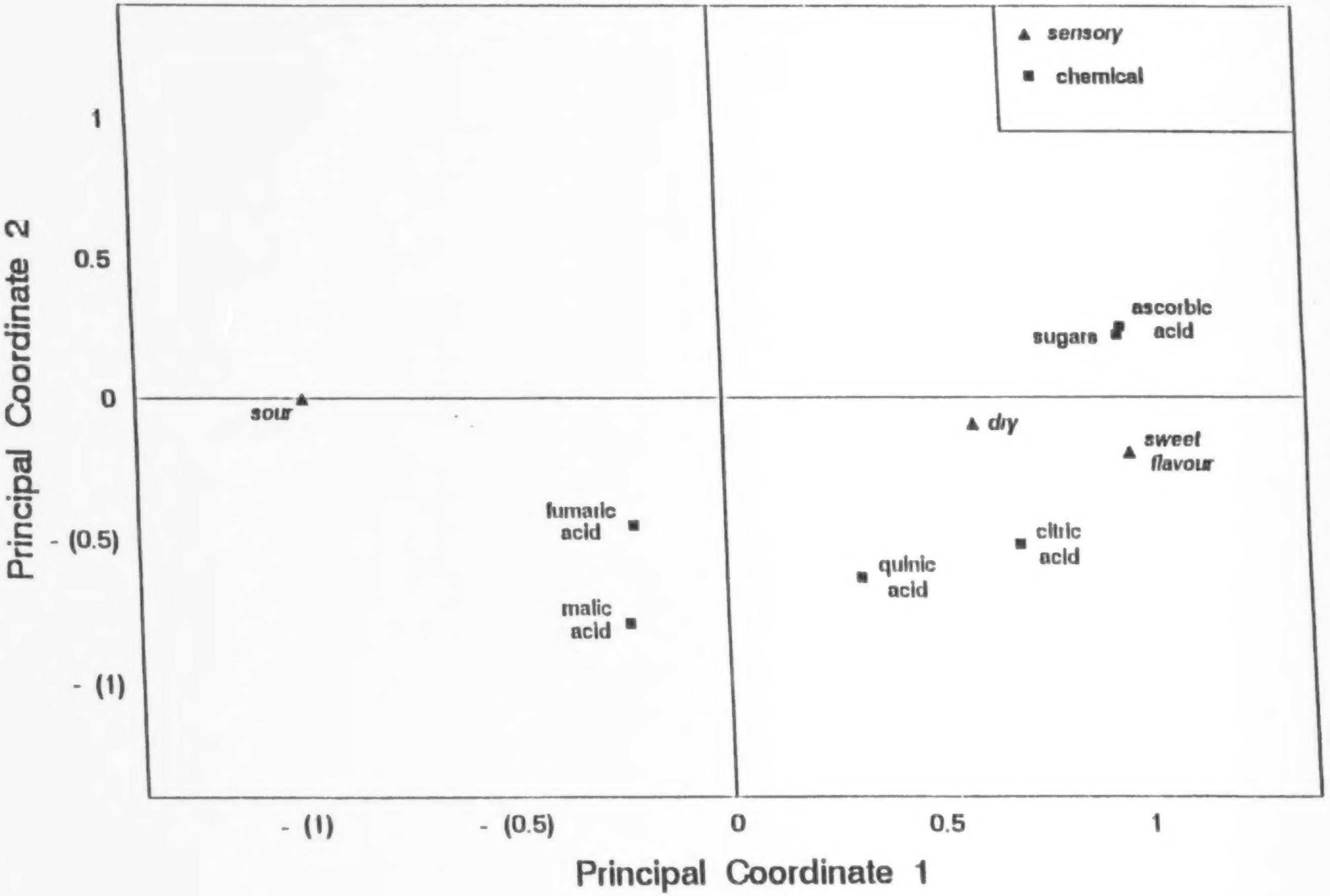

Figure 7.3. GPA correlation plot of instrumental and sensory taste and oral sensation of processed saskatoon berry juice.

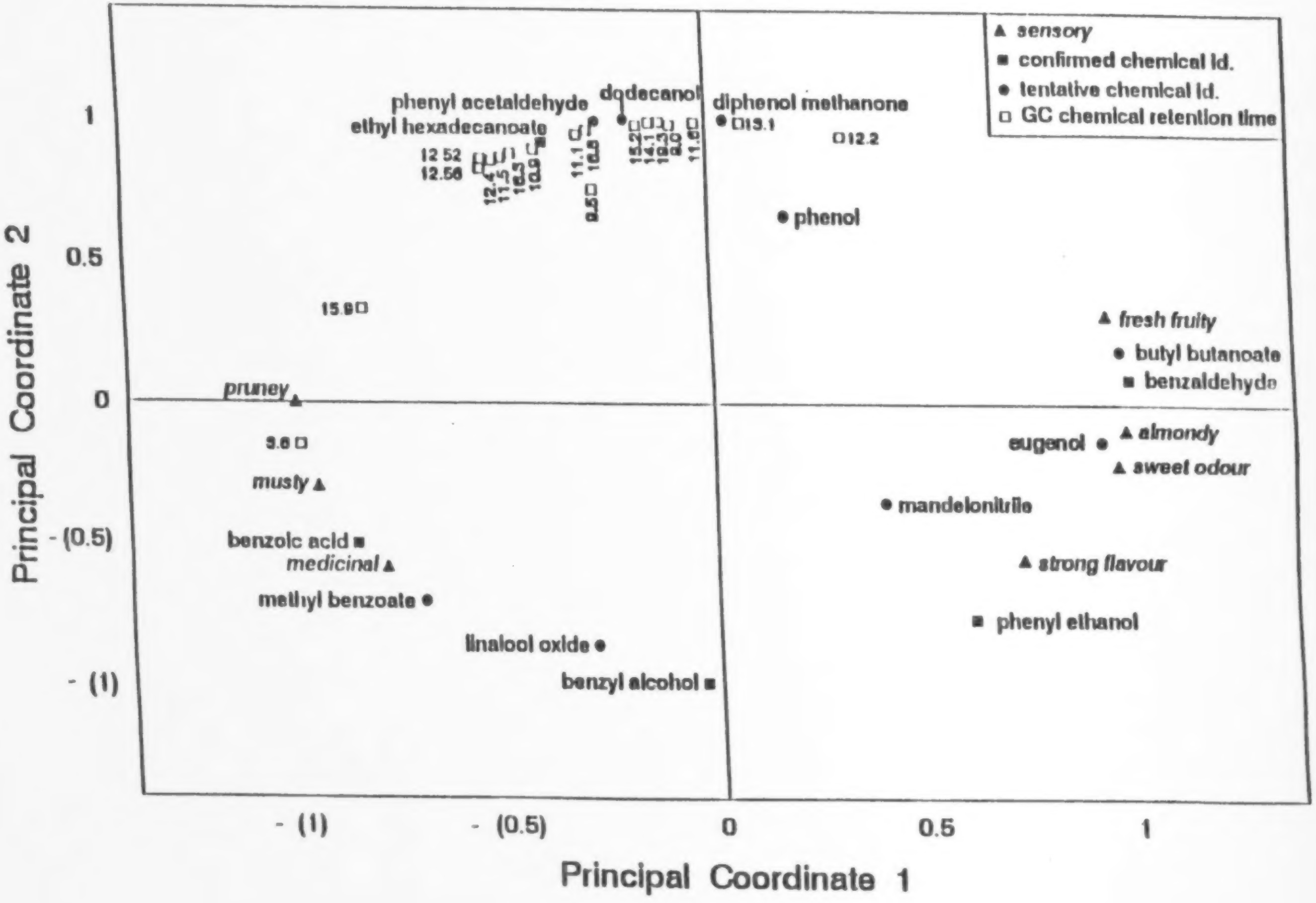

Figure 7.4. GPA correlation plot of instrumental (GC SE54 column) and sensory odour and flavour strength of processed saskatoon berry juice.

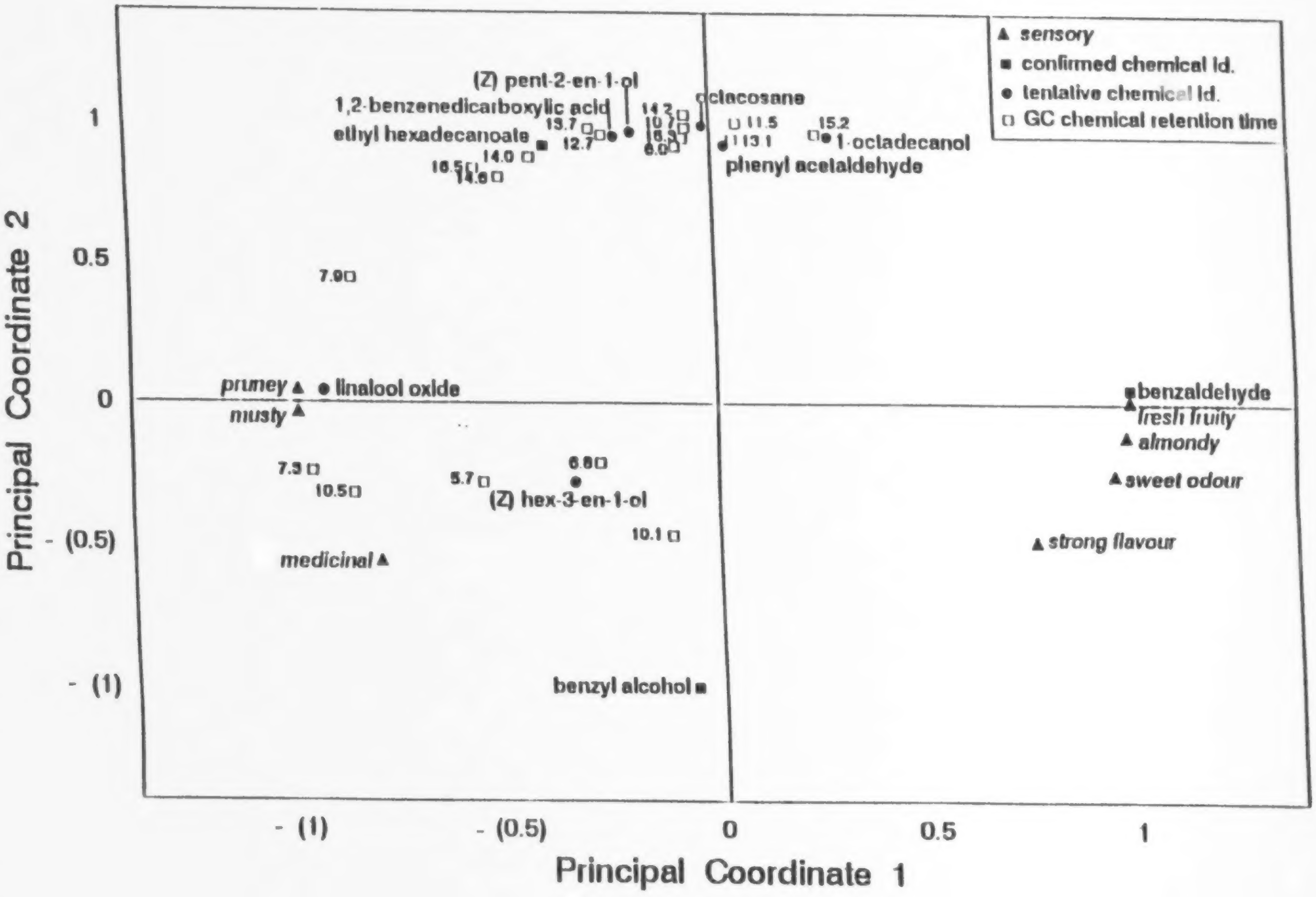

Figure 7.5. GPA correlation plot of instrumental (GC Supelcowax 10 column) and sensory odour and flavour strength of processed saskatoon berry juice.

odour. Thus, the sensory perception of sweet odour may result from the presence of both benzaldehyde and phenyl ethanol.

The SE54 column analysis (Figure 7.4) shows fresh fruity at the positive end of PC1 a little more towards the positive end of PC2 than for the Supelcowax 10 column analysis (Figure 7.5). The SE54 column analysis also identified a GC peak, not found with the Supelcowax 10 column, which appeared next to fresh fruity odour on the plot. This peak was tentatively identified as butyl butanoate (p=0.81) which is characterised as fresh, sweet, and fruity (Flath and Forrey, 1977).

The SE54 column identified two peaks, eugenol (clovish) and mandelonitrile (unstable precursor of benzaldehyde), which appear in the same quadrant as almondy and sweet odour. These volatile constituents have not previously been reported as contributing to almondy or sweet odour (Dravnieks, 1985).

Strong flavour is another sensory characteristic associated with the positive end of PC1, but it also correlates to the negative end of PC2. The correlation plots (Figures 7.2, 7.4, and 7.5) show that benzaldehyde, butyl butanoate, eugenol, phenyl ethanol, mandelonitrile, benzyl alcohol, and sugars may all contribute to the strong flavour of the juices.

Medicinal is positioned at the negative ends of both PC1 and PC2 as are benzoic acid and methyl benzoate. Neither compound has been reported in the literature as being medicinal; benzoic acid is not a significant odour compound (Croteau and Fagerson, 1968) and methyl benzoate is pungent, heavy, sweet, and floral (Idstein and Schreier, 1985). In the present research benzyl alcohol was used as the standard for medicinal for the QDA panel (chapter five). In both correlation plots benzyl alcohol appears at the negative end of PC2. Benzyl alcohol was determined in increasing quantities as follows: C < H < P < HP juices (chapter three) and thus correlates strongly to PC2. Medicinal odour related to PC2 in a similar fashion as benzyl alcohol. In the present research the sensory perception of medicinal in H juice may have been masked by strong fruity, almondy, sweet notes (chapter five). Medicinal odour also related to the negative region of PC1 where C, P, and HP juices were located. Thus, benzyl alcohol is probably a major volatile constituent contributing to medicinal odour of processed saskatoon juices.

Musty and pruney odours appear together in the negative region of PC1 in both odour/flavour strength correlation plots. The GC SE54 column analysis showed two peaks and the GC Supelcowax 10 column analysis showed four peaks in the vicinity of musty and pruney. Only one of these peaks (from the GC Supelcowax 10 column) was tentatively identified as linalool oxide. Linalool oxide has been characterised as earthy (MacLeod and Pieris, 1983). Linalool oxide is found in the negative regions of PC1 on both odour/flavour strength plots. The two plots show differing positions for linalool oxide with respect to PC2; SE54 column plot location at -1 on PC2 and Supelcowax 10 plot location close to 0 on PC2. The location of the musty descriptor also moves slightly towards the negative end of PC2 in Figure 7.4 as compared to Figure 7.5. Thus, linalool oxide may contribute to the musty characteristic of saskatoon juices. Further identification of GC peaks is required, especially for those with retention times of 3.6 and 15.9 min for the SE54 column, and 5.7, 6.8, 7.3 and 10.5 min for the Supelcowax 10 column in order to determine whether these volatile constituents contribute to pruney and/or musty notes in saskatoon juice.

GPA resulted in a large number of GC peaks from both the SE54 and Supelcowax 10 column analyses which correlated to the positive end of PC2 but did not relate to specific sensory descriptors. Most of these peaks had long retention times and were high molecular weight compounds. Researchers have shown that many of the character impact components of fruit flavours are highly volatile, low molecular weight compounds (Hirvi and Honkanen, 1982). Thus, these high molecular weight compounds may not contribute significantly to the predominant flavour characteristics of saskatoon juice.

The results of this study indicated that GPA was a useful tool for the identification and correlation of instrumental data with sensory data for four processed saskatoon juices. Relationships found included: prune brown colour with Hunterlab 'L' and 'b' values; dark cherry colour with Hunterlab 'L' and 'a' values; sweet taste with sugars; dryness with quinic acid; almondy with benzaldehyde; sweet odour with benzaldehyde and phenyl ethanol; fresh fruity with butyl butanoate; medicinal with benzyl alcohol; musty with linalool oxide;

and strong flavour with a combination of benzaldehyde, phenyl ethanol, butyl butanoate, and benzyl alcohol. Further work is required to identify GC peaks in saskatoon juices which may correlate with pruney odour.

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Chapter 8 General Discussion and Recommendations

Saskatoon berries are currently being cultivated in Alberta at a rate which will permit the development of value-added products (Hausher, 1988). The berries must be juiced to manufacture wines, jellies, and syrups. Methods used for commercial juicing of other fruits include cold, hot, and enzyme assisted. Studies on the juicing of saskatoon berries have been limited to laboratory scale heat and enzyme extractions which evaluated juice yield, acids, and sugars (Wolfe and Wood, 1971; Mazza et al., 1978; Mazza, 1979). Aroma studies have shown that 45% of headspace volatiles were lost when berries were juiced (Mazza, 1980) and that benzaldehyde was a major aroma component (Mazza and Hodgins, 1985). Majak et al. (1981) found cyanogenic glycosides, including amgydalin, present in saskatoon berries. Amygdalin enzymatically produces benzaldehyde when fruit tissues are disrupted (Swain et al., 1992). Thus, the distillation of Mazza and Hodgins (1985) may have created benzaldehyde as an artefact of the volatile analysis. Sensory studies on the quality of saskatoon juice are limited to observations that homogenized berries smelled warm, sweet, and almondy (Mazza and Hodgins, 1985) and that saskatoon jelly had a weak flavour (Mazza, 1979).

This study addressed the need for: 1) complete instrumental and sensory data on the effects of processing methods on saskatoon juice quality; and 2) the establishment of relationships between objective and sensory measurements of saskatoon juices. The processing methods investigated on a pilot plant scale included: cold (C); pectinase enzyme (P); hemicellulase and pectinase enzymes (HP); and hot (H) juicing of saskatoon berries. Instrumental data were obtained for yield, colour, sugars, acids, and volatile constituents of each of the four juice treatments. The sensory attributes of the saskatoon juices were determined by both trained QDA and naive consumer FCP panels. FCP results were compared to QDA results using GPA. GPA was also investigated as a technique to relate instrumental and sensory data.

Chapter 2 (a preliminary trial) compared yield, soluble solids, pH, and five volatile constituents in C and H processed saskatoon juices. The differences among juices found in Chapter 2 were similar to those in Chapter 3 which also included P and HP methods.

In Chapter 3, juice yields from HP, P, and H methods were significantly greater (4-16%) than that from C method. These pilot plant results differ from those of Mazza et al. (1978), Mazza (1979), and Mazza (1980) which showed no differences in yields of enzyme versus cold pressed saskatoon juices. Thus, the effect of enzymes on yield when using a screw-type pulper finisher (chapter three) was greater than when using a hydraulic press (Mazza et al., 1978; Mazza, 1979, and Mazza, 1980). The clarity of the juices varied; HP had a large amount of particulate matter and H juice was clear. Particulate matter in juices was reduced with centrifugation but yields were decreased during the process. The most cost effective saskatoon juicing process would vary depending on the cost of enzymes, availability of equipment, and desired characteristics in the end product. H processing would achieve the highest yield of juice for clear jellies and HP would be best for cloudy syrups.

HunterLab colour differences showed that C and P juices were significantly lighter than HP and H juices; C and P juices were significantly more yellow than H juice; and all juices were similar in red values. The lightening and yellowing of juice colour is indicative of oxidative enzymatic browning (Moyer and Aitken, 1980). Heat, as in the H process, can inactivate the enzymes (Dimick et al., 1951) and preserve the typical purple/red anthocyanin colour. The addition of acid (ie. citric) to juice also stabilizes anthocyanin pigments (Green and Mazza, 1986). Thus, high yielding HP juice which has browned slightly maybe suitable for syrup production with the addition of acid.

All four saskatoon juices contained similar quantities of fructose but H juice had a higher glucose content than the other three juices. All four treatments also contained malic, citric, quinic, fumaric, and ascorbic acids; the latter three have not been previously reported in saskatoon berries. Acid contents were similar among the treatments except for fumaric acid which was significantly higher in P juice than in C, HP, and H juices. Neither the ion-pairing (Simpson, 1982) nor the ion-exchange (Coppola and Starr, 1986) HPLC methods provided a complete organic acid profile of saskatoon juice. Quinic acid was not resolved using ion-exchange HPLC. Using the ion-pairing technique, quinic acid was well resolved but coeluted with tartaric acid.

Results obtained from the ion-exchange method indicated that tartaric acid, if present, is found in very low quantities. Therefore, the peak from ion-pairing analysis is probably primarily quinic acid. The ion-pairing technique did not resolve malic acid very well and resulted in lower malic acid concentrations than previously reported in saskatoon juice (Wolfe and Wood, 1971). Further studies using both HPLC techniques are required to obtain more accurate quantifications of organic acids in saskatoon juice.

Using a SE54 column, GC analysis of solvent extracts of saskatoon juices resolved more peaks (30) than with a Supelcowax 10 (23). More low molecular weight constituents were resolved using the Supelcowax 10 column than the SE54 column. Use of the Supelcowax 10 column may be advantageous for flavour analysis as low molecular weight volatiles frequently have significant aroma impact in fruits (Hirvi and Honkanen, 1982). Of the 48 GC peaks resolved in the processed juices, 18 were identified (chapter three). The most significant differences among the juice treatments were found in three aromatic compounds: benzaldehyde, benzyl alcohol, and benzoic acid. Benzaldehyde levels differed significantly among the treatments with H>HP>P>C juice (Table 3.6). Benzyl alcohol levels differed significantly among the treatments with HP>P>H>C juice (Table 3.6). Benzoic acid levels in HP and P juices were similar and significantly higher than in C juice which was also higher than in H juice (Table 3.5). Further experiments (chapter four) determined the effect of the presence of seeds, water, heat, and time on the levels of benzaldehyde, benzyl alcohol, and benzoic acid in juices. Some of these experiments were preliminary trials conducted prior to experiments in Chapters 2 and 3 and others were carried out later with the limited fruit remaining. As a result, the juicing techniques used in each of the experiments (chapters two, three, and four) differed in batch size and equipment. However, the GC analyses were conducted according to the methods outlined in Chapter 3. The differences in aromatic compounds among the experiments were far larger than would be expected as a result of differences in batch size and/or equipment. The findings confirmed the presence of low levels of benzaldehyde, benzyl alcohol, and benzoic acid in seedless saskatoon juice. Although Tuttas (1977) concluded that free benzaldehyde was absent in intact cherry tissues, the current research findings are consistent with those of other researchers (Honkanen and Pyysalo, 1976; Hirvi et al., 1981; Salles et al., 1988) who determined benzaldehyde, benzyl alcohol, and benzoic acid in cloudberries, cranberries, and apricots. These aromatic constituents may result from phenylalanine metabolism (Pyysalo, 1976) as described in Chapter 1. The presence of seeds and water increased levels of benzaldehyde, benzyl alcohol and benzoic acid in saskatoon juices. Benzyl alcohol formation was highest initially (15°C) and decreased slightly (to 85°C) while benzaldehyde and benzoic acid formation were highest in the 45-75°C range. Results (chapter four) also indicated that two separate enzyme systems were involved in the production of benzaldehyde and benzyl alcohol. The enzymatic reaction which produces benzaldehyde from the cyanoglucoside amygdalin, present in the seeds, has been documented (Wong, 1989) The enzymatic reaction which yields benzyl alcohol is not known. The benzoic acid and benzaldehyde contents of saskatoon juices appear related; benzaldehyde is oxidized to benzoic acid. The results of Chapters 2, 3, and 4 indicate that the four processing methods affect the levels of over 40 volatile constituents in the resulting juices. If the benzaldehyde, benzyl alcohol, and benzoic acid peaks are removed from the chromatograms, the total volatile peak area of C > P > HP > H juice. These overall decreases in volatile constituents may have a significant effect on the quality of saskatoon juice from each treatment.

A trained QDA panel (n=6) developed a lexicon of eleven characteristics for the four processed saskatoon juices (Chapter 5). Differences among the juices were found for ten of the eleven of the lexicon terms. Juices from C, P, HP processing were all described as prune brown in colour and cloudy; having musty and pruney odours; as well as sour taste and a dry oral effect. Enzyme processed juices (P and HP) differed significantly from C juice by slight almondy and strong medicinal notes. H processed juice was significantly different from the other three juices and was described as black cherry in colour and clear; having fresh/fruity, almondy, and sweet odours; sweet taste; and a dry oral effect. Overall, C juice had a significantly weaker flavour than P, HP, and H juices. These results indicate that, although the concentrations of many volatile constituents were lower in P, HP, and H juices than C juices (chapters 2 and 3), the large increases in aromatic compounds (Chapters 2 and 3) contributed to the stronger flavours of P, HP, and H juices than C juice. Consumer products, such as wines, jellies, and syrups, made from H juice would thus have a different flavour profile than those made from C, P, and HP juices.

Two consumer FCP panels (2 X n=24) evaluated the four saskatoon juices using vocabularies of descriptive terms which were unique to each panelist (Chapter 6). Data from the two FCP panels as well as from the QDA panel (Chapter 5) were analysed by generalised Procrustes analysis (GPA). The three consensus configurations from each panel showed similar differentiation of the four juice treatments. The QDA panel results determined greater differentiation among the juice treatments than the FCP panels. This was expected as the QDA panelists were intensely trained and used defined descriptors for saskatoon juices. The terms used by the consumer FCP panelists closely matched those of the QDA panel. The QDA panel was able to differentiate the juices using the terms sweet odour and sour taste; however, the FCP panels were not. The two FCP panels showed good experimental reproducibility and the GPA showed that 82-83.6% of the variation among the treatments was explained by the consensus configurations. The QDA panel consensus plot explained 95.2% of the variation among the juice treatments. The process for the FCP panel took 5 d to complete while the QDA panel process took 3 mo. Guy et al. (1983) and Steenkamp and van Trijp (1987) also found consumer FCP panels to be effective for describing differences among whisky and meat, respectively. Quarmby and Ratkowsky (1988) found FCP of fish spoilage unsuccessful due to the inability of panelists to develop vocabularies to describe the products. Success of the present research in utilising FCP may be due to the fact that the juices were fairly easy to describe in simple common terms and the samples were quite different, especially C and H juices. Hedonic responses to the juices, from the FCP panels, indicated that H juice was liked more than the others. Characteristics such as clarity, red colour, fresh, fruity, almondy, and sweet were all attributes of H juice that may have contributed to its high hedonic score. The low hedonic scores given by the FCP panel to P and HP juices may have been the result of the medicinal note. Thus, high yielding enzyme processed juices may have an objectionable medicinal note which may need to be masked by the creation (as in H process) or addition of other aroma compounds. The present FCP study indicates that use of a consumer FCP panel may be a fast, and thus economical, way of obtaining information on differences among products as well as indicating product acceptability. FCP appears to provide good results when the products being tested are relatively easy to describe and the differences among the products are reasonably large.

The final objective of the present study was to determine whether relationships among objective and subjective data obtained in Chapters 3 and 5 could be established. GPA is a multivariate technique which translates, rotates, and scales individual data matrices to arrive at a consensus configuration which minimizes the distances between each individual configuration. Although GPA has been used for sensory (Gower, 1975; Guy et al. 1983; Steenkamp and van Trijp 1987) or analytical data (Liardon et al. 1984), there are no published reports of GPA being used to combine sensory and analytical data matrices together in one analysis. This thesis used the unique approach of determining sensory and instrumental relationships through a combined GPA. Analytical data from Chapter 3 and sensory data from Chapter 5 were averaged to obtain one value for each analytical measurement, the first matrix, and each sensory characteristic, the second matrix. Since the two matrices contain different measurements (instrumental versus sensory) the GPA of variance tables cannot be interpreted. However, the correlation plots, which relate instrumental measurements and sensory characteristics to the two principal coordinates, can be used visually to establish relationships among the two data sets for the juices. Relationships established from the consensus plots, some supported by other researchers, included: prune brown colour with 'L' and 'b' values; dark cherry colour with 'L' and 'a' values; sugars with sweetness; almondy with benzaldehyde (Dravnieks, 1985); sweet odour with benzaldehyde (Dravnieks, 1985) and 2-phenyl ethanol (Stevens et al., 1970); fresh/fruity with butyl butanoate (Flath and Forrey, 1977); medicinal with benzyl alcohol; musty with linalool oxide (MacLeod and Pieries, 1983); and strong flavour with a combination of benzaldehyde, 2-phenyl ethanol, butyl butanoate, and benzyl alcohol. Although numerical correlation values were not obtained using GPA, the technique was useful in focusing the identification of GC peaks according to those which were adjacent to sensory descriptors on the plot. GC peaks next to fresh/fruity and musty characteristics on the plot were subsequently identified as butyl butanoate and linalool oxide, respectively. Literature quoted above confirmed the relationship between these two volatiles and the sensory descriptors. Further work needs to be undertaken to identify peaks near the pruney characteristic to establish a relationship for the one sensory characteristic in saskatoon juices which was not accounted for. Other peaks in the GC chromatograms should also be identified as sensory descriptors are frequently the result of combinations of volatile constituents.

In order to economically produce high quality saskatoon products such as wines, jellies, and syrups an appropriate method for juicing the berries must be selected. This thesis determined many significant instrumental and sensory differences among saskatoon juices processed by four commercial methods. Hot processing of saskatoon berries produced a high yield of clear juice with colour, odour, and taste characteristics which were liked by consumer panelists. Hemicellulase and pectinase enzyme processing produced a very high yield of cloudy saskatoon juice with a slightly brownish colour which may be made more red/purple by the addition of acid (anthocyanin pigment is more red/purple at lower pH's) and an undesirable medicinal note which may be masked by the creation or addition of other flavours.

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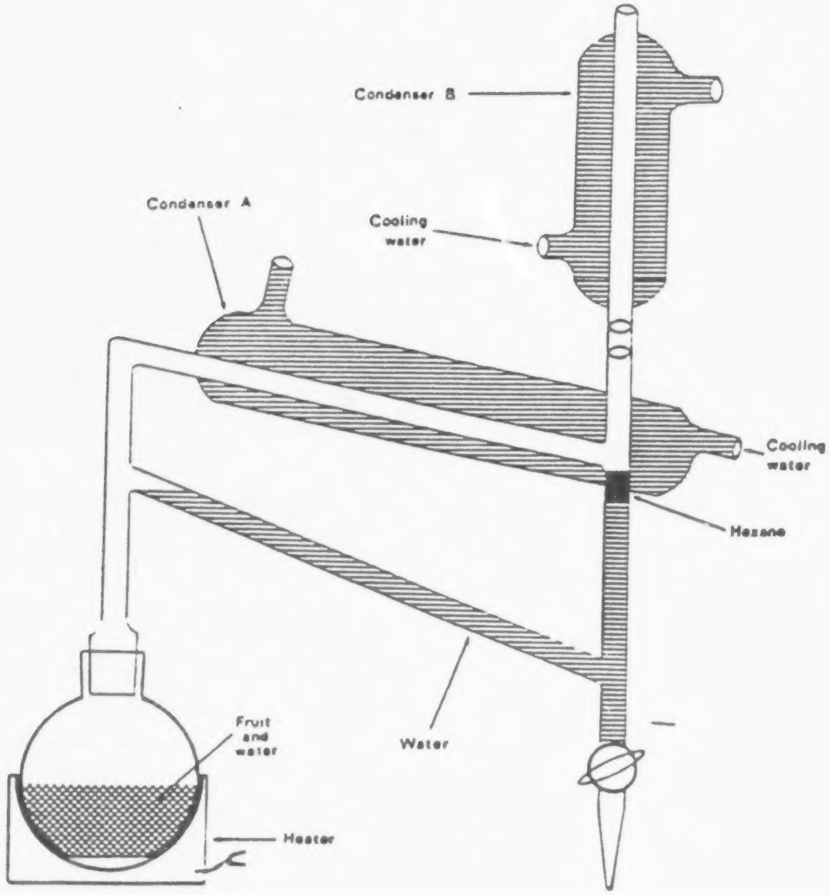
Appendix 1

Distillation apparatus for Chapter 3 / experiment 3 (Mazza and Hodgins, 1985)


Condenser B
Condenser A
Cooling water
Cooling water
Hexane
Fruit and water
Water
Heater

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